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TEXTILE RESEARCH JOURNAL

VOL. XVI, No. 12 December, 1946

Relationship Between Molecular Configuration and Tensile Properties of Protein Fibers*

G. C. Nutting, M. Halwer, M. J. Copley, and F. R. Senti

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IT has been demonstrated that globular proteins can be changed into the extended, quasi-crystalline molecular form typical of natural fibrous proteins and that the change produces a notable increase in fiber tenacity [1, 2, 3]. To show more fully the effect of altered molecular configuration on mechanical properties of protein fibers, data are presented in this paper on stress-strain characteristics—strength, elongation, stiffness, toughness, and elasticity—of one globular protein (ovalbumin) and the fibrous proteins keratin, silk fibroin,‡ and collagen, in corresponding configurational states. Ovalbumin was chosen as the most suitable globular protein, because of the especial ease and completeness with which its molecules can be given a linear configuration. Comparison of its tensile properties with those of keratin was particularly sought since diffraction patterns of oriented globular proteins and stretched (*beta-*) keratin prove the near identity of their spatial arrangement. Tensile tests were made on keratin in its normal, somewhat folded (*alpha-*) configuration; in its fully extended, *beta*-keratin form; and after supercontraction as disoriented keratin. Collagen and fibroin filaments were tested in the ordinary, fibrous condition, and also after disorientation by contraction.

Materials and Experimental Procedures

Ovalbumin, prepared from the whites of fresh eggs by the method of Sørenson and Høyrup [4], was mixed with half its weight of distilled water, kneaded into a plastic mass, extruded through a 13 mil die, and denatured by boiling ½ hour in distilled water. Orientation was achieved by stretching the denatured filaments in steam. The stretched fibers were relaxed by immersing several hours in water at 25°C. No formaldehyde or other chemical after-treatment was given. Fiber diameters ranged from 150 to 400 microns.

Wool, mohair, and horsehair * were the keratin fibers used. The wool was a high-grade 62s blend, received in the form of top. The mohair ranged in diameter from 23 to 34 microns. The horsehair used for most of the tests was black, with diameter 90 to 170 microns, averaging 125. White, violin-bow horsehair was generally unsuitable, since most of the filaments had a low cystine content and contracted spontaneously in boiling water, owing probably to

* Parts of this paper were presented at meetings in Philadelphia of the American Chemical Society, June 13, 1945, and The Fiber Society, February 28, 1946.

† One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, United States Department of Agriculture.

‡ Hereafter the term "fibroin" will be used.

* The authors would like to thank Dr. Werner von Bergen of the Forstmann Woolen Co., Passaic, N. J., for supplying the wool sample; Dr. A. C. Whitford of S. Stroock & Co., Newburgh, N. Y., for supplying the mohair; and the Frederick H. Cone Co., New York, for supplying the horsehair.

photochemical damage while still on the animal. Selected, undamaged hairs, however, were used for the load-elongation experiments reported below, the black hair being too short for this purpose. All the keratin samples were washed successively in alcohol, ether, and water.

The collagen was U.S.P. size 5–0 surgical gut. It consisted of a single strand with a twist of two turns per inch. It had received no hardening or tanning treatment but had been stretched during manufacture.

The fibroin was U.S.P. size 000 suture silk. Each strand consisted of three plies twisted together, each ply being composed of about 100 filaments 10 microns in diameter. For these tests the strands were separated into their component plies.

Horsehair was converted into the *beta*-keratin form by stretching to relative length * (r.l.) 1.70 in steam and "set" by steaming 3 hours in the stretched state to stabilize it against water at room temperature. After the fibers had been cut out of the stretching clamp, they were relaxed by soaking for several hours in water at room temperature. During relaxation, the fibers contracted until the final r.l. was 1.63. To determine the effect of steaming in the stretched state, horsehair was converted into the *beta*-keratin form without steaming by stretching to r.l. 1.50 in cold water. Since these fibers were not set, it was necessary that they be tested without being relaxed. The results thus obtained gave a measure of the maximum strength obtainable from horsehair in the *beta*-keratin form, since the deleterious effect of steam and the disorientation produced by relaxing were eliminated.

Wool was put into the *beta*-keratin form by mounting small parallelized bundles in clamps and stretching to r.l. 1.50 in cold water and was then set by steaming 3 hours while stretched. The fibers were relaxed in water at room temperature. The final r.l. was 1.41.

Mohair was stretched to r.l. 1.80 in steam and set by steaming one hour. The stretched fibers were relaxed as above. The final r.l. was 1.67. Mohair was selected because its length, uniformity of diameter, and freedom from medullation make it particularly suitable for single-fiber tests. Thus it can be compared with horsehair more readily than can wool, whose nonuniform diameter makes it inapt for single-fiber tests. A comparison between horsehair and mohair was necessary to establish the influence, if any, of fiber diameter on the properties studied.

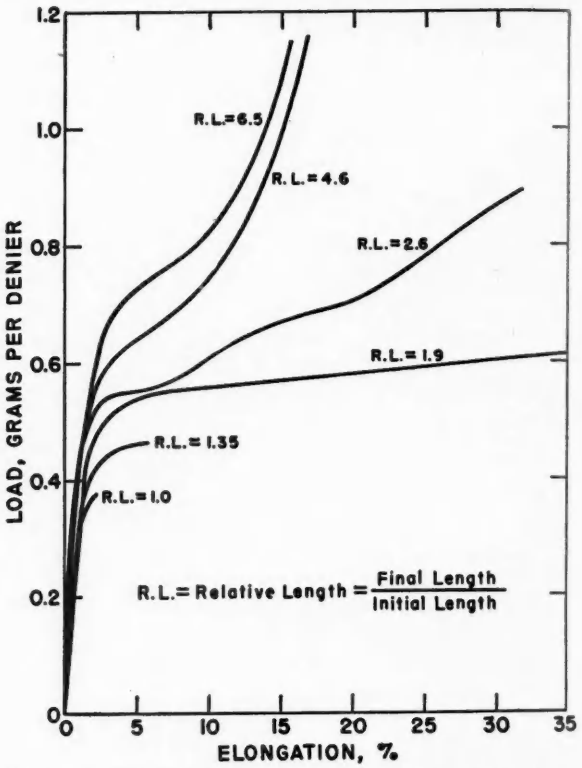

FIG. 1. *Effect of orientation on load-elongation curves of ovalbumin.*

Wool and horsehair were supercontracted by modifications of methods developed by Speakman [5] and Astbury [6]. Wool was supercontracted by stretching parallelized bundles 50 percent in cold water, steaming 3 minutes while stretched, releasing and steaming one hour further. The final r.l. was 0.75. Horsehair was stretched 40 percent in cold water, then boiled 3 minutes in an 0.05*M* phosphate buffer of pH 8.0 (measured at 25°C). The fibers were now released from the clamp, boiled ½ hour in the buffer, washed in distilled water, and dried. The average supercontraction was 21 percent, corresponding to r.l. 0.79. When stretched horsehair was steamed, as was the wool, supercontraction was nonreproducible. The variability was found to be a pH effect and was eliminated by substitution of buffer solution for steam.

Collagen and fibroin were tested in two stages of orientation. The higher stage was that of the ma-

* Relative length (r.l.) = final length/initial length. In the experiments described there was usually concurrent change in molecular orientation and fiber length. Sometimes the fiber was stretched and subsequently contracted to less than its unstretched length, making inappropriate such conventional descriptions as "percent stretch" or "draw ratio." To indicate the dimensional changes we have chosen to use the somewhat cumbersome term "relative length." By this is meant the ratio of the final fiber length to the initial length in a particular series of operations.

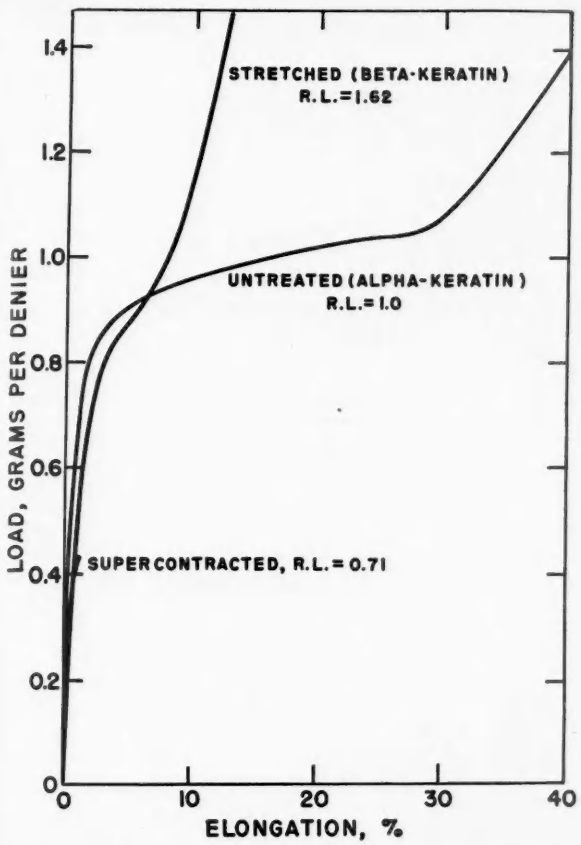

FIG. 2. *Effect of orientation on load-elongation curves of keratin (horsehair).*

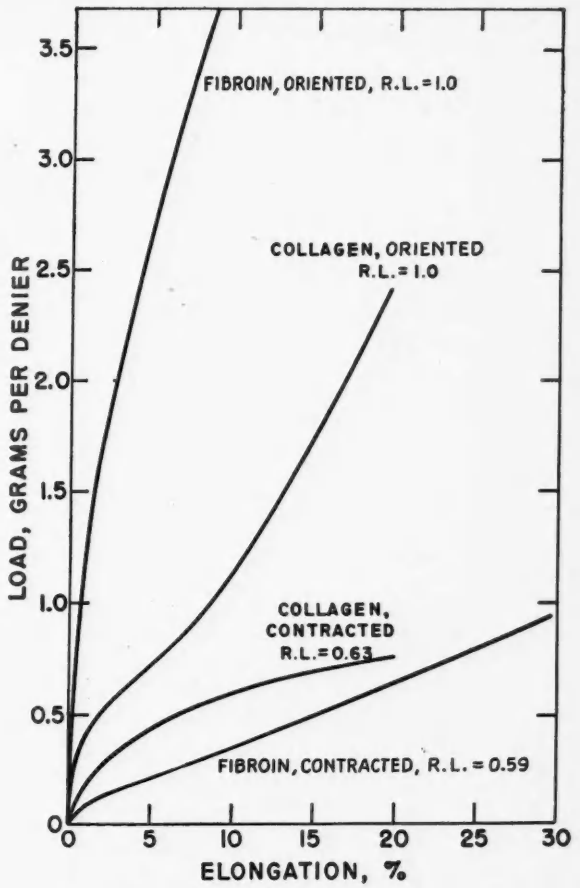

FIG. 3. *Effect of orientation on load-elongation curves of fibroin and collagen.*

terials as received. The samples could be stretched only a little before breaking, except following drastic chemical treatment, and their orientation could not be much improved. A lower stage of orientation was induced by contraction. This was easily done for collagen by immersing the fiber in water at 70°C for 30 seconds, an average contraction to r.l. 0.54 being obtained. Fibroin was contracted to r.l. 0.54 by immersing it for 90 seconds in 8.42 *M* HCl at room temperature. It was washed in water, followed by 1 percent sodium acetate, and again by water. The concentration of acid required to produce contraction was remarkably critical. Thus, 8.00*M* HCl produced no contraction, whereas a concentration of 8.42*M* produced a contraction of 46 percent. Different acids showed different minimum concentrations below which contraction did not occur, but all agreed in the sharpness of the minimum. A more complete account of this phenomenon is in preparation.

In all cases in which a change in orientation was produced as a result of dimensional change, control samples were prepared in which changes in orientation were minimized by keeping the fibers clamped at their original length. In every other respect, the controls received the same treatment as the experimental samples.

To determine to what degree the disorientation process can be reversed, the contracted forms of horsehair and collagen were restretched. Horsehair was contracted to r.l. 0.74, stretched in steam to r.l. 1.78, steamed 3 hours while stretched, then relaxed in water. The final r.l. was 1.68. Thus, the reoriented horsehair was at approximately the same extension as the samples which had been stretched without prior supercontraction. Catgut, contracted to r.l. 0.54, was stretched to r.l. 1.19 in water at 50°C. After drying in the clamps, the fibers were relaxed in the free state to constant length in water at room temperature. Their final r.l. was 0.95.

Appropriate control samples were prepared for the reoriented horsehair, but not for the reoriented collagen. Instead, the same controls which had formerly been prepared for the contracted samples were

TABLE I. EFFECT OF ORIENTATION ON TENSILE PROPERTIES OF OVALBUMIN FIBERS

Relative length	Tenacity (g./den.)	Wet/dry tenacity ratio	Flexibility
1.0	0.60	0.28	0.77
2.4	0.89	0.44	0.81
4.2	1.14	0.54	0.37
5.8	1.44	0.52	0.23

used, on the assumption that a brief exposure to water at 50°C was unlikely to produce appreciable change in fibers which had already been exposed at 70°C.

In all cases, the effectiveness of the various treatments in changing molecular orientation was determined from x-ray diffraction patterns of the product. Diffraction results are discussed below.

Tenacities were measured with a Scott IP–2 testing machine at 70°F and 65 percent R.H. Values reported are the mean of ten tests. Samples were permitted at least 16 hours to reach moisture equilibrium before testing. Deniers of the mohair fibers were determined microscopically and for all other fibers by weighing a measured length. Single fibers of horsehair, mohair, collagen, and ovalbumin were tested. A modified A.S.T.M. bundle test, similar to that of Peterson *et al.* [7], was used for wool. For fibroin, strands consisting of about 100 filaments with a twist of five turns per inch were used. Wet tenacities were determined immediately after soaking the fibers in distilled water at room temperature for one-half to one hour. Knot strengths were measured by tying a simple overhand knot in the fiber and then determining the breaking strength. The ratio of the knot strength to the straight-pull strength is reported as "flexibility." *

Strength and Flexibility Measurements

Results of tenacity tests are given in Tables I and II and may be summarized as follows. For ovalbumin the dry tenacity increased regularly from 0.6 g./den. at r.l. 1 to 1.44 g./den. at r.l. 5.8. (By r.l. 3, incipient resolution of the diffraction pattern into arcs demonstrated partial orientation of peptide chains parallel to the direction of drawing, and at r.l. 6 the *beta*-keratin structure was fully developed.) On stretching, the flexibility appeared to increase at first, then decreased markedly, so that at r.l. 5.8 the knot tenacity was only 0.3 g./den. A similar relation between stretch, tenacity, and flexibility was observed by McMeekin *et al.* [8], who worked with quinone-hardened casein filaments. This is notable because it implies molecular orientation even though

* The knot test gives a measure of the diminution in strength when a fiber is bent over a small mandrel. The test is complex, involving elements of tension, compression, and shear, and is correspondingly difficult to analyze, but, especially when the knot strength is divided by the ordinary tensile strength, it provides a useful index of fiber brittleness.

TABLE II. EFFECT OF ORIENTATION ON TENSILE PROPERTIES OF NATURAL PROTEIN FIBERS

Test No.	Material and treatment	Relative length	Tenacity (g./den.)	Wet/dry tenacity ratio	Flexibility
1	Horsehair, untreated (*alpha*)	1.00	1.46	0.87	0.62
2a	Horsehair, stretched, steamed (*beta*)	1.63	1.42	0.65	0.13
2b	Horsehair, control for 2a (*alpha*)	1.00	1.22	0.79	0.62
3a	Horsehair, supercontracted	0.79	0.63	0.44	0.78
3b	Horsehair, control for 3a (*alpha*)	1.00	1.27	0.70	0.58
4a	Horsehair, reoriented (*beta*)	1.68	1.12	0.62	0.33
4b	Horsehair, control for 4a (*alpha*)	1.00	1.15	0.78	0.77
5	Horsehair, stretched, not steamed (*beta*)	1.50	2.06	—	0.27
6	Wool, untreated (*alpha*)	1.00	1.08	0.71	—
7a	Wool, stretched, steamed (*beta*)	1.41	1.08	0.66	—
7b	Wool, control for 7a (*alpha*)	1.00	0.97	0.72	—
8a	Wool, supercontracted	0.75	0.46	0.39	—
8b	Wool, control for 8a (*alpha*)	1.00	1.03	0.70	—
9	Mohair, untreated (*alpha*)	1.00	1.44	—	0.87
10a	Mohair, stretched, steamed (*beta*)	1.67	1.89	—	0.24
10b	Mohair, control for 10a (*alpha*)	1.00	1.32	—	0.87
11	Collagen, oriented	1.00	2.82	0.75	0.48
12a	Collagen, contracted	0.54	1.05	0.60	0.76
12b	Collagen, control for 12a and 13	1.00	2.43	0.82	0.49
13	Collagen, reoriented	0.95	1.77	0.59	0.54
14	Fibroin, oriented	1.00	3.88	0.71	0.79
15a	Fibroin, contracted	0.54	1.44	0.67	1.00
15b	Fibroin, control for 15a	1.00	3.45	0.79	0.93

TABLE III. EFFECT OF ORIENTATION ON MECHANICAL PROPERTIES OF PROTEIN FIBERS, DEDUCED FROM LOAD-ELONGATION CURVES

Material and treatment	Relative length	Ultimate elongation, %	Tenacity, g./den.	Stiffness* g./den.	Toughness index,† g./den.	Elasticity‡ %
Ovalbumin, denatured	1.0	2.2	0.36	17.1	0.004	2.2
Ovalbumin, stretched	1.35	8.4	0.43	5.8	0.02	3.0
Ovalbumin, stretched	1.9	33.8	0.64	2.2	0.11	4.0
Ovalbumin, stretched	2.6	32.6	0.92	2.8	0.15	3.0
Ovalbumin, stretched	4.6	17.8	1.16	6.6	0.11	3.3
Ovalbumin, stretched	6.5	15.3	1.17	7.7	0.09	3.4
Horsehair, supercontracted	0.71	3.5	0.42	16.5	0.008	2.4
Horsehair, untreated (*alpha*)	1.0	40.0	1.40	3.5	0.28	4.1
Horsehair, stretched (*beta*)	1.62	12.9	1.54	11.9	0.10	2.9
Fibroin, contracted	0.59	32.9	0.98	3.0	0.17	—
Fibroin, oriented	1.0	9.1	3.65	40.5	0.17	—
Collagen, contracted	0.63	20.5	0.76	3.9	0.08	—
Collagen, oriented	1.0	21.9	2.66	12.1	0.30	2.6

* Stiffness = T/E, where T = tenacity in g./den. and E = ultimate elongation, expressed as a fraction of the original length.

† Toughness index = $\frac{TE}{2}$.

‡ Elasticity = elongation up to yield point of load-elongation curve.

the spatial order of the casein molecules is not perfect enough to give a fiber diffraction pattern comparable to that of ovalbumin. Wet tenacity of ovalbumin filaments rose with orientation from 0.17 to 0.75 g./den.

The keratins showed remarkably similar behavior. Thus *alpha*-keratin horsehair (1.22 g./den., test 2b, Table II) when stretched and steam-set became *beta*-keratin with strength 1.42 g./den., wet/dry tenacity ratio 0.65, and flexibility 0.13 (test 2a). When the stretched horsehair was contracted to r.l. 0.79 (test 3a), orientation as measured by diffraction was largely removed, the tenacity dropped to 0.63 g./den. and the wet/dry tenacity ratio to 0.44, while flexibility increased to 0.78. Impairment of *alpha*-keratin, produced by a 3-hour steam treatment analogous to that employed to stabilize the *beta*-keratin configuration, amounted to 0.24 g./den. (tests 1 and 2b). The maximum average strength recorded was 2.06 g./den., obtained by stretching horsehair to r.l. 1.5 in water and letting the specimen dry while clamped (test 5). The usual practice of relaxing in cold water could not, of course, be applied. Reorientation of contracted horsehair (test 4a) raised the strength and decreased the flexibility as expected, but the degree of orientation achieved was considerably less than in the initial preparation of *beta*-keratin by stretching *alpha*-keratin. Data on wool (tests 6–8) and mohair (tests 9 and 10), while less complete, show similar effects of orientation on tensile properties. The unexpectedly small increase in tenacity when wool was converted to the *beta*-keratin form (Table II, tests 7a and 7b) might at first be taken to indicate that the rupture of wool under stress is due to slippage of cells rather than to slippage of protein chains. It seems more likely that the considerable difficulty experienced in making bundles for testing from stretched wool was responsible for the low strength.

Oriented collagen, on contraction to r.l. 0.54, underwent a change in tenacity from 2.43 to 1.05 g./den. (tests 12a and 12b). As with keratin, only partial reorientation was attained, with strength rising to 1.77 g./den. at r.l. 0.95 and flexibility diminishing from 0.76 to 0.54. Analogous results on collagenous material have been obtained by Valko, Wöhlisch, and others [9].

The tenacity of fibroin changed from 3.45 to 1.44 g./den. on contraction to r.l. 0.54 in 8.4*M* HCl (tests 15a and 15b). Especially noteworthy was the high initial flexibility, 0.93, since the degree of orientation was evidently somewhat higher than in even the best oriented specimens of collagen, keratin, and ovalbumin.

Load-Elongation Properties

Figure 1 shows typical load-elongation curves for ovalbumin fibers at several stages of orientation, while Figure 2 presents the curves for horsehair, and Figure 3 for collagen and fibroin. In Table III average values for the tenacity and ultimate elongation are listed, together with the stiffness, the toughness in-

dex, and the elasticity.* The last three properties are also derived from the load-elongation curves and have been commended by Smith [10] for the characterization of fibrous materials. An IP–2 testing machine was used to obtain the curves. Owing to the wide range in denier and strength of the various fibers, it was impracticable to keep the rate of loading constant. For horsehair it varied from 1.0 to 2.7 g. per denier per minute; for mohair, 4.9 to 8.0; wool, 1.6 to 1.8; ovalbumin, 0.7 to 1.8; collagen, 1.3 to 3.1; contracted fibroin, 1.6; oriented fibroin, approximately 10. Specimens ranged from 7 to 18 inches in length, depending principally upon the extensibility of the material tested and the capacity of the machine.

Ovalbumin. Shape changes of the ovalbumin curves with orientation are noteworthy. Unoriented fibers (r.l. 1) were incapable of supporting the stress required to produce flow during the test. The extensibility was therefore only a few percent and consisted almost entirely of elastic elongation. A small amount of orientation (r.l. 1.35) strengthened the fiber sufficiently to permit flow, with a resultant significant increase in elongation at break. At r.l. 1.9 elongation was maximal, but there was little reinforcement during the flow process, indicating that the principal units of flow were micelles consisting of large groups of molecules. At r.l. 2.6, elongation was still high and there was distinct reinforcement, which became pronounced at r.l. 4.6. This implies that the chief unit of flow in the latter case was the individual molecule. The molecules straighten out, pack more closely, and interact more strongly among themselves, with consequent reinforcement. At r.l. 6.5 the shape of the curve was much the same as at r.l. 4.6. There was a sharp drop in elongation between r.l. 2.6 and 4.6, corresponding to the change in the flow mechanism and to the partial straightening of chains produced by stretching.

Comparison of Table III with Table I shows that, except for the slightly stretched fibers, there is good correspondence between ultimate elongation and knot-strength flexibility. The large decrease in flexibility between r.l. 2.4 and 4.2 (Table I) was paralleled by the decrease in elongation between r.l. 2.6 and 4.6 (Table III). Flexibility seems to be the more sensitive function of orientation.

Tenacity values in Table III tend to be a little lower than those in Table I, a result to be expected from the greater length of the specimens employed. Both the stiffness, which is related to the pliability of fabric woven from the fiber, and the toughness index, which is a measure of the fiber's capacity to absorb work without breaking, were affected strongly by orientation. At r.l. about 2.5, elongation, flexibility, and toughness were at a maximum, while stiffness was nearly minimal. Tenacity and wet strength were improved by further stretching, but only at the expense of other desirable properties. The (Hooke's law) elasticity was practically independent of the state of orientation.

Keratin (Horsehair). The stress-strain curves for horsehair are similar to those for ovalbumin. Disoriented (supercontracted) horsehair, like unoriented ovalbumin, was too weak to flow and broke after only slight elongation. The normal (*alpha*-) configuration resembled ovalbumin of r.l. 2.6 in having high extensibility, but showed more distinct reinforcement. Stretched horsehair (*beta*-keratin) resembled highly stretched ovalbumin in the general form of its curve and in its sharply diminished elongation at break.

On comparing Table III with Table II (tests 1 and 2a) it is evident that the large decrease in elongation that accompanied *alpha*- to *beta*-keratin conversion was matched by the drop in knot-strength flexibility. The analogy with ovalbumin is clear. The same is true of the contrast between the low elongation of supercontracted keratin and its high flexibility ratio (test 3a, Table II). Like ovalbumin, the elasticity of horsehair did not change much with the state of orientation. It is doubtful whether the differences observed are significant, in view of the difficulty of fixing the yield point exactly. The well-known long-range reversible elasticity to which *alpha*-keratin fibers owe so much of their value was not, of course, manifested in these tests.

Collagen. Oriented collagen gave a curve of the same general form as horsehair and oriented ovalbumin. Contracted collagen, however, showed no reinforcement, from which it appears that the unit of flow was the micelle. Since there was no definite yield point, no estimate of elasticity was made. The elongation of collagen was not much changed by contraction. The increase in elongation which should result through folding of the linear molecules during contraction was offset by the weakening effect of disorientation, causing the fiber to break before the molecules were fully extended. The better oriented form had a much greater toughness index than the contracted form.

* Quantitative definitions of stiffness, toughness, and elasticity are given in Tabie III (footnotes).

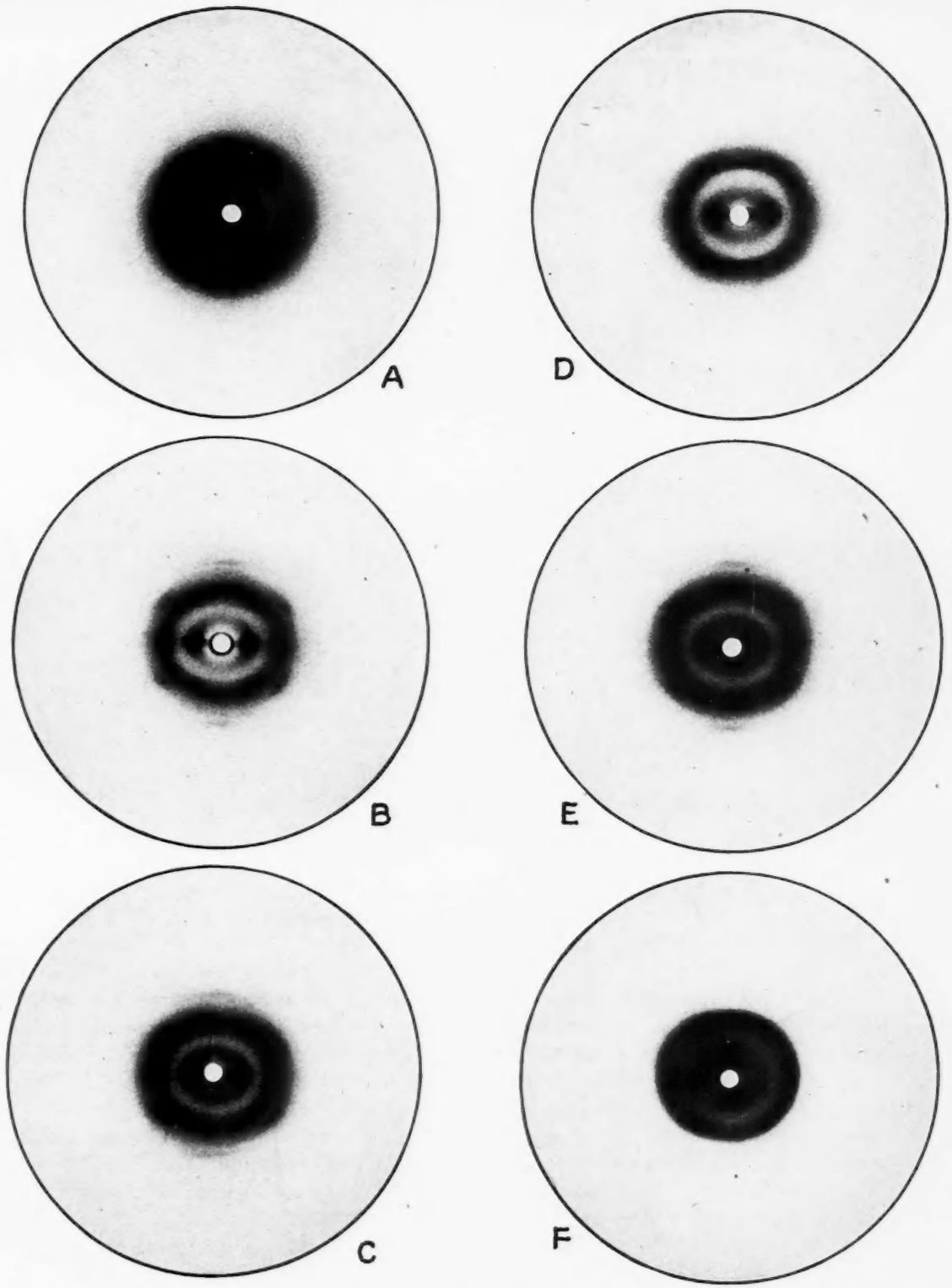

FIG 4. *Diffraction patterns of ovalbumin (A, B, C) and keratin (D, E, F) filaments. (A) Ovalbumin, r.l. = 1; (B) r.l. = 10.5; (C) oriented filament after boiling 8 hours in distilled water; (D) unoriented keratin (horsehair), α-structure; (E) β-structure, r.l. = 1.7; (F) supercontracted, r.l. = 0.7. Ni-filtered Cu radiation; 2.5-cm. specimen to film distance. Fiber axis vertical.*

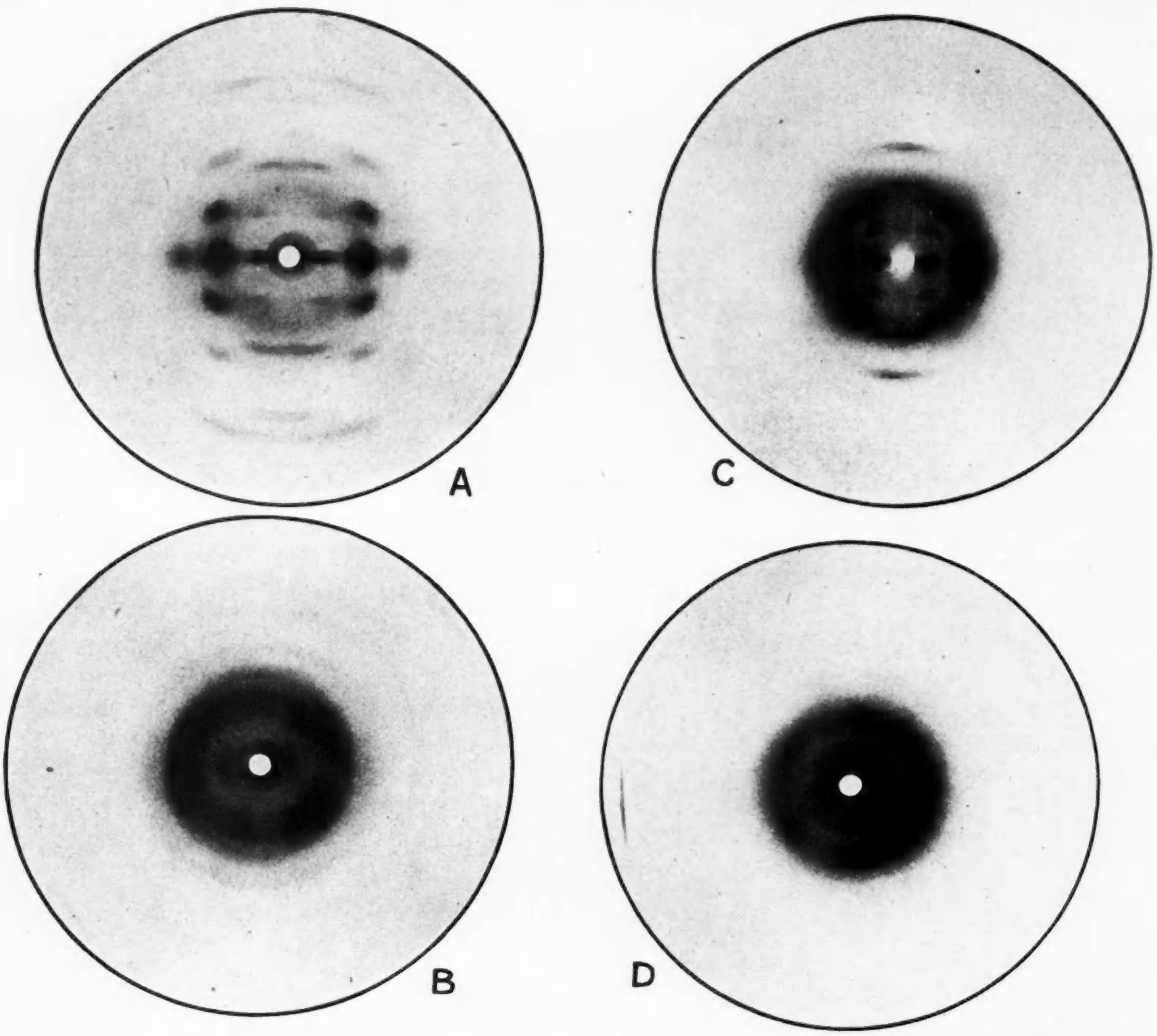

FIG. 5. *Diffraction patterns of fibroin (A, B) and collagen (C, D) filaments. (A) Oriented fibroin, r.l. = 1; (B) contracted fibroin, r.l. = 0.5; (C) oriented collagen, r.l. = 1; (D) contracted collagen, r.l. = 0.5. Ni-filtered Cu radiation, 2.5-cm. specimen to film distance. Fiber axis vertical.*

Fibroin. Neither oriented nor contracted fibroin showed a definite yield point. The elongation (9.1 percent) found for oriented fibroin in this experiment was lower than the values usually given in the literature. Appel [11], for example, gives the range of elongation of fibroin as 13–20 percent. The combination of low elongation with high tenacity produces a high stiffness value, but, because of the limited elongation, the toughness index is relatively low.

Diffraction and Birefringence Observations

Primary indication of changes in configuration and spatial order was by means of x-ray diffraction. Figures 4A, B, and C reproduce diffraction patterns of ovalbumin filaments. Figure 4A is the pattern of a heat-denatured, unstretched preparation (r.l. 1). The sharpness of the rings indicates a fair degree of crystallinity, and the distribution of the diffracting units with respect to the fiber axis is random. Stretching in steam to r.l. 10.5 produced a high degree of orientation parallel to the direction of stretch (Figure 4B), but, judging from the sharpness of the arcs, there was little change in crystallinity. The crystallites or diffracting regions of highly oriented ovalbumin are surprisingly stable toward boiling water. Figure 4C is the pattern of a fiber, originally at r.l. 10.5, that had been boiled 8 hours in distilled water. Although the arcs are lengthened slightly and are somewhat less distinct, it is apparent that the

crystallites have suffered little damage. Previous experience [3] indicates that such treatment with boiling water would result in longitudinal contraction of the fiber of roughly 35 percent and in a corresponding large loss in tensile strength. Both effects must have developed principally in noncrystalline regions.

Figures 4D, E, and F show horsehair keratin in the *alpha*- form (r.l. 1), *beta*- form (r.l. 1.62), and supercontracted form (r.l. 0.71). The practical identity of highly oriented ovalbumin and *beta*-keratin in their crystallite configuration is obvious. Reference to the stretch given the two fibers (r.l. 1.62 and 10.5) illustrates the far greater ease of orientation of the keratins compared with ovalbumin. Lacking the numerous cystine cross-bonds of keratin, molecules and micelles of ovalbumin fibers stretched in steam flow easily and only a minor part of the work of stretching is effective in securing fiber orientation. Supercontraction removed most but not all orientation. In Figure 4F there is slight concentration of intensity in the inner (9.7 Å.) ring in the equatorial direction. The pattern corresponds to ovalbumin stretched in steam to r.l. approximately 1.5. Crystallinity is less, however, for the pattern is more diffuse and the 3.7 Å. (outer) ring is not resolved. No counterpart of the *alpha*-keratin structure has yet been reported for ovalbumin or other globular protein.

Figure 5A is the fiber pattern of fibroin; Figure 5B the pattern for fibroin contracted in 28*M* formic acid to r.l. 0.45. Figure 5C is the diffraction pattern of oriented collagen; Figure 5D is that of the same material contracted to r.l. 0.63 by brief immersion in hot water. As with *beta*-keratin, contraction of both fibroin and collagen greatly diminished the degree of orientation, without changing the structure of the crystallites.

Birefringence also measures molecular orientation, summing effects of both amorphous and crystalline components, and showing particular sensitivity for low and otherwise undetectable degrees of orientation. Table IV gives the birefringence of the six protein fibers with which we have worked. *Beta*-keratin has little higher birefringence than *alpha*-keratin, but its value is much greater than that for supercontracted keratin. While the birefringence of fibroin is considerably reduced on contraction, in both stages of orientation it is uniquely high among protein fibers. Birefringence of collagen is lowered almost 80 percent on contraction; its maximum value is the least of any of the fibers sufficiently oriented to give a well-defined fiber diffraction pattern. Orientation was in no case completely removed by contraction, for positive birefringence was measured in all fibers but unstretched ovalbumin. Residual birefringence in the contracted ovalbumin fiber whose diffraction pattern is shown in Figure 4C was 6.2×10^{-3}. This fiber, of initial r.l. 10.5, shrank ⅓ during 8 hours' boiling.

TABLE IV. BIREFRINGENCE OF PROTEIN FIBERS

Fiber	Relative length	Birefringence
Keratin		
Horsehair, *alpha*	1.0	8.6×10^{-3}
Horsehair, *beta*	1.7	10.4
Horsehair, contracted	0.76	2.0
Wool, *alpha*	1.0	7.6
Wool, *beta*	1.5	9.8
Wool, contracted	0.82	3.5
Mohair, *alpha*	1.0	8.2
Mohair, *beta*	1.8	8.6
Fibroin, normal	1.0	73.0
Fibroin, contracted	0.37	33.0
Collagen, oriented	1.0	5.9
Collagen, contracted	0.55	1.2
Ovalbumin *	1.0	0.0
Ovalbumin *	2.0	5.0
Ovalbumin *	6.0	12.0

* Data from reference [3], p. 205.

Discussion

Keratin fibers differ from the others in that they are cellular. In comparing them the assumption was necessarily made that the mechanical and rheological properties measured depend predominantly on intracellular material. Woods [12] had previously shown that the elasticity behavior of a single cortical cell of wool closely approximated that of the complete wool fiber. If there is an effect of cell structure on tensile properties, little evidence for it emerges from this study.

Speakman [5] has attributed supercontraction in the keratins to the breakdown of disulfide cross-links and electrostatic bonds, and permanent set to the breakage of these bonds with subsequent formation of new bonds. If this is correct, then the changes in properties observed on converting keratin to the *beta*- or to the supercontracted form could be due to chemical alteration as well as to changes in the state of orientation. Preliminary analyses for cystine and cysteine [13], however, revealed no significant differences in the amount of these substances in set and supercontracted keratin fibers as compared with the *alpha*-keratin controls. Work on this aspect of the problem is continuing. It should be noted in this connection that Patterson *et al.* [14] found that breaking

the cystine cross-links in wool, by reduction with thioglycolic acid, produced little change in the dry tenacity. It is difficult to judge the effect of the broken electrostatic bonds, since the proportion of these required to be broken in order to bring about supercontraction or permanent set is unknown. Moreover, it is to be expected that most of them would re-form when the stress which caused them to break was removed.

As was stated in the introductory paragraph, among the dozen globular proteins that have been given linear form demonstrable by x-ray diffraction, ovalbumin is outstanding in the extent to which molecular orientation and crystallinity can be produced in it. In the present work it has been shown that maximum orientation raises dry and wet tenacity, but not to the extent that they are comparable with fibroin and collagen; and in achieving maximum tenacity through maximum orientation other important fiber qualities—elongation, flexibility, toughness—are largely sacrificed. Dry tenacity of high-orientation ovalbumin is about the same as that of keratin fibers. Wet tenacity is appreciably lower, but may be made equal by cross-linking with quinone or formaldehyde. Oriented ovalbumin has not, in our experience, shown any tendency to relax into a regularly coiled molecular configuration, and thus lacks the longe-range reversible extensibility that is so usefully characteristic of the keratins. Fibroin and collagen are distinguished from ovalbumin by their flexibility at high orientation. From this difference, particularly, we infer a low effective molecular weight for ovalbumin [3]. Experiments have been begun to measure the effective molecular weight or polymerization degree of the orienting units in this and other globular proteins.

Summary

1.Fibers compared were heat-denatured ovalbumin stretched up to 500 percent in steam but not hardened; keratin (wool, mohair, horsehair) in the *alpha-*, *beta-*, and supercontracted states; fibroin in its normal, oriented form, and after contraction by 8.4*M* HCl; collagen (surgical gut) when oriented and after contraction in hot water.

2. X-ray diffraction patterns demonstrate the near identity of the spatial configuration of *beta*-keratin and highly stretched ovalbumin, and of supercontracted keratin and slightly stretched ovalbumin. Contraction of all the natural fibers is attended by serious loss of orientation, but lateral order or crystallinity in the diffracting units is little affected.

3. Positive birefringence shown by all the fibers is considerably reduced on contraction but does not vanish, demonstrating a fair degree of residual orientation in the contracted fibers. Conversion of *alpha-* to *beta*-keratin produces only a small increase in birefringence.

4. Wet and dry strength increase markedly with molecular orientation. Flexibility, as measured by knot tenacity, decreases.

5. Changes in load-elongation properties of the fibers with orientation are shown. From load-elongation curves, values for elasticity, ultimate elongation, stiffness, and toughness have been tabulated.

6. Tensile properties of *beta*-keratin horsehair and maximally oriented and crystallized ovalbumin are remarkably similar. Tenacity increase in both fibers on maximum orientation is more than offset by unfavorable changes in toughness, stiffness, and elongation.

Acknowledgment

The authors take pleasure in acknowledging the assistance of Elizabeth W. Johnson in performing the experiments described.

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Oxygen Balance in Hydrogen Peroxide Bleaching

S. A. Simon and Arthur Drelich

A contribution of the Research Division, Chicopee Manufacturing Corporation

An experimental procedure for securing an oxygen balance is given using a typical bleach formula with cotton. Data are graphically presented showing decomposition of peroxide, evolution of oxygen, and consumption of oxygen as functions of time. Oxygen balance is suggested as a parameter for evaluating bleaching systems. It is indicated that oxygen balance data in conjunction with data obtainable by other known tests can provide an insight into bleaching otherwise not obtainable.

Introduction

In conventional bleaching practice cotton is subjected to one or more of the following purification procedures prior to the actual bleaching: desizing, demineralizing, and alkaline extraction. As a result of these operations a relatively high order of cellulosic purity may be obtained before any bleaching action is initiated. However, some of the remaining impurities are sufficiently colored [1] so that their removal is essential where a high degree of whiteness is desirable. The attainment of this whiteness is a principal function of the bleaching process.

The two principal commercial bleaching processes are commonly called the chlorine and peroxide processes, respectively; both are oxidative in nature. Because peroxide in its commercially available state (as hydrogen peroxide or sodium peroxide) is relatively expensive, it becomes imperative to secure the maximum possible bleaching effect with a minimum of peroxide. Since there exist in cotton a number of materials capable of consuming the bleaching agent, it is essential to know the functions of these materials and the oxygen distribution among them. The determination of this oxygen balance has received little attention in the literature on hydrogen peroxide bleaching [2]. Consequently, in this paper a method will be described for determining oxygen balance, and the type of data secured from a typical bleaching solution of hydrogen peroxide will be illustrated. The method has general application but the data and interpretations presented apply specifically to the cottons, solutions, and ratios of solution to cotton described. The described ratios resemble most closely those occurring in the kier-bleaching process.

The exact mechanism by which bleaching takes place is still open for discussion in spite of the great amount of study expended on the kinetics of the decomposition of hydrogen peroxide solution alone. The responsibility for this situation lies in the heterogeneous nature of the bleaching process, as well as the lack of certainty of the actual cotton constituents participating. However, the daily production of commercially satisfactory bleached cottons attests to the existence of a practical over-all knowledge of bleaching.

Control in the bleaching of cotton is secured through adjustment of the peroxide concentration, pH, total alkalinity, time-temperature relationships, the ratio of solution to cotton, and by the addition of certain catalysts. By suitably adjusting these conditions the properties of the bleaching solution and of the resultant product may be varied over a useful range.

Theory

The conventional bleaching system may be considered as consisting of two distinct parts: first, the free peroxide solution in which oxygen is being released independently of the cotton at a distance from it and, second, that portion of the solution in close contact with the colloidal cotton surface. In the latter instance the hydrogen peroxide may be decomposed either catalytically to liberate oxygen or by a chemical

reaction with cotton constituents in which oxygen is consumed.

The common practice of determining the rate of peroxide decomposition, by titrating the bleaching solution, reveals nothing of the mechanism of bleaching. It will not differentiate the oxygen evolved by the solution in catalytic contact with the cotton from the oxygen being evolved by the free solution. Nor will this analysis determine the oxygen actually consumed in reaction with the cotton and its impurities. Since it is reasonable to believe that only the oxygen which is actually consumed by the reaction bleaches the cotton, it is apparent that attempting to determine the course of a bleaching operation merely by analyzing the solution for its peroxide content is futile. The situation is rendered more complex when it is realized that the various cotton constituents may be consuming oxygen at different rates and that some of the oxidation taking place may be in no way related to the whitening process. While it is possible that oxygen being consumed by the cotton serves a useful function other than bleaching, it is known that under such conditions as high alkalinity and temperature excessive consumption of oxygen is indicative of undesirable reactions [3]. Consequently, it is also desirable to know whether it is the cellulose or the impurities which are consuming the oxygen.

Since it is not possible to bleach satisfactorily simply by introducing oxygen gas into the system, it is concluded that the oxygen evolved from the free bleach solution does not contribute to the whitening function and represents an undesirable side reaction to the whitening. Moreover, the oxygen evolved catalytically is not involved in the whitening function. Therefore, only the oxygen consumed can be utilized for the obtaining of the bleaching effect. Furthermore, since the oxygen consumed may be reacting either with the cellulose or the noncellulosic substances, or both, the actual predominating course of the consumption reaction is a major factor along with the total amount of oxygen actually consumed. From considerations along this line it is possible to apportion the oxygen consumed to the following: (*a*) cellulose, (*b*) colorless impurities associated with cotton, and (*c*) colored substances present in cotton. Of these three possible consumption reactions, only the last is definitely desirable. The first is actually detrimental and may result in real injury if allowed to proceed too far. The second path does not directly improve the whiteness of the cotton although it may have some other commercial value. The third is beneficial only if the reaction products are rendered colorless or soluble in the bleaching solution. Therefore, not only is it important to direct the largest possible proportion of the peroxide into consumption, but the course of the consumption reactions must be led along favorable paths. Adequate methods for the detection and estimation of damage to cellulose [4] and for the measurement of degree of whiteness of cotton are in common use [5]. In this paper only the estimation of oxygen consumption will be considered.

An over-all oxygen balance indicates only three possible points of oxygen distribution: first, oxygen remaining as undecomposed hydrogen peroxide; second, oxygen evolved from the solution as gaseous oxygen; and, third, oxygen consumed by the cotton in chemical combination. This statement may be summed up by the equation

$$T = R + E + C, \qquad (1)$$

where T represents the total initial oxygen introduced as H_2O_2, R represents the oxygen remaining in solution as undecomposed peroxide, E represents the oxygen evolved as gaseous oxygen, and C represents the oxygen consumed by the cotton.

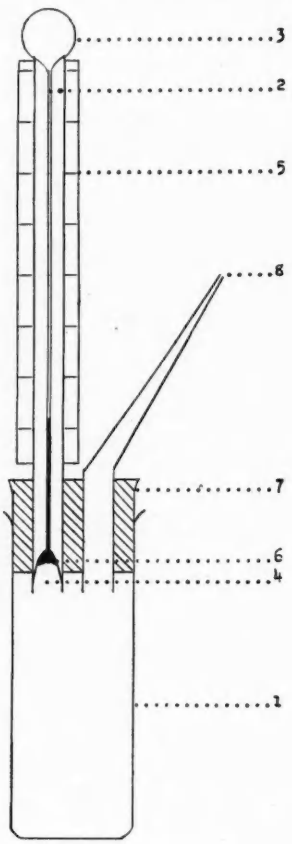

FIG. 1. *Volumescope.*

T, R, and E can be determined experimentally. Since $T - R$ is the amount decomposed, D, then it follows that $R = T - D$. Substituting this value for R in equation (1), the following equation is obtained:

$$C = D - E. \qquad (2)$$

Experimental Plan

The plan of the experiment in determining consumption, C, is based on the derived equation of oxygen balance, $C = D - E$. Values for D, the amount decomposed, and E, the amount evolved, are determined experimentally. D is determined by introducing known amounts of hydrogen peroxide into a bleaching system and then following the decomposition of the peroxide in conventional manner by titration. E is determined by manometrically measuring the gaseous oxygen evolved. C is then determined as the calculated difference between D and E.

Because of the complications associated with the withdrawal of bleaching solution from a pressure vessel, it was not possible to measure both the evolved oxygen and the undecomposed oxygen data as functions of time from the same specimen. A volumescope was so designed that the oxygen evolved during the progress of bleaching was measured continuously. In this way one specimen gave evolution data over the entire time range discussed herein. For the decomposition data a bleaching system, comparable to that in the volumescope, was placed in a test tube fitted with an air condenser. At a selected time interval the amount of oxygen remaining was determined by titration. The amount decomposed (D) is calculated as the difference between the original total oxygen (T) and that remaining (R). Each point on the decomposition curve, showing the amount decomposed as a function of time, thus represents an individual specimen. In this way data were secured on the behavior of the bleaching solution, alone, in the presence of commercially bleached cotton sliver, and in the presence of raw cotton sliver.

To illustrate the heterogeneity of the bleaching mechanism, data on the behavior of three different ratios of bleaching solution to cotton were also obtained.

Experimental Procedure

The bleaching solution is an 0.13N hydrogen peroxide solution containing 5.0 grams of sodium sili-

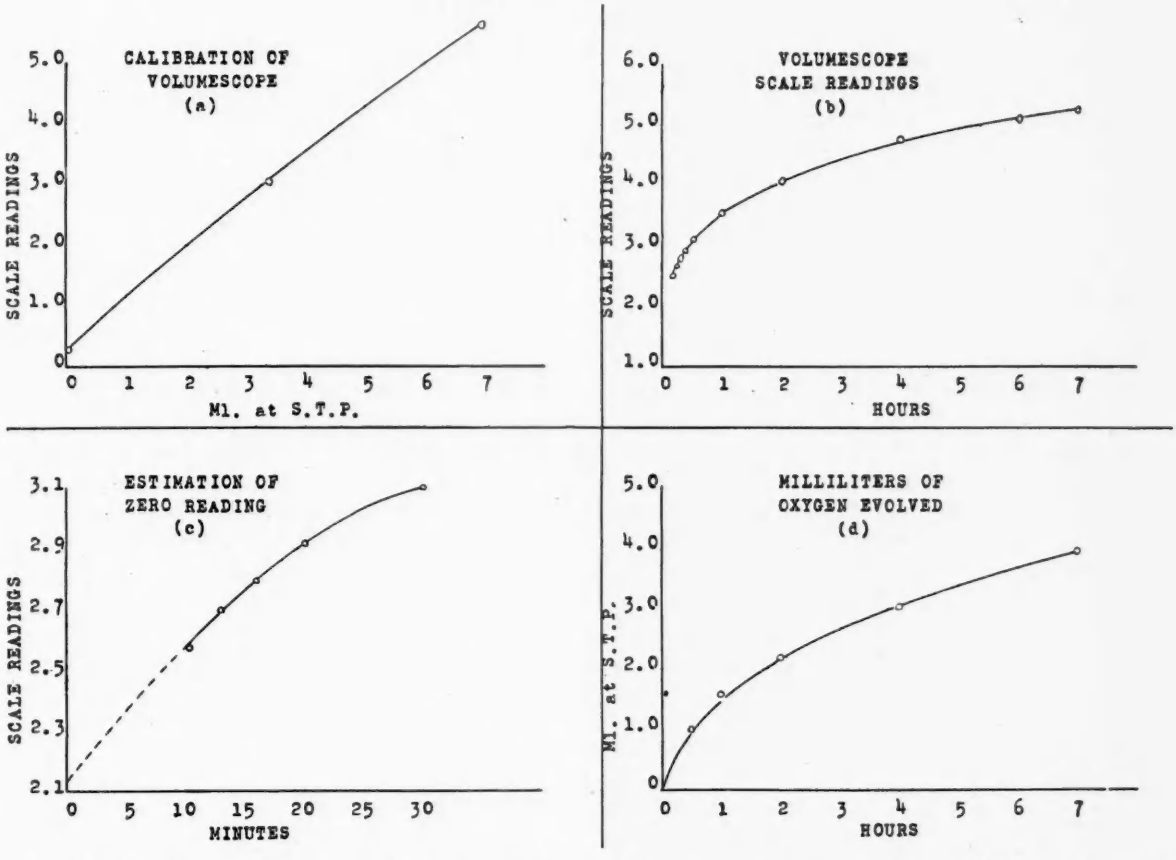

FIG. 2. *Graphs showing calibration and conversion of the observed scale readings.* (*S.T.P.* = *standard temperature and pressure.*)

cate and 0.50 gram of sodium hydroxide per liter. The peroxide used is the commercially available 30-percent grade. The sodium hydroxide is reagent grade, whereas the silicate is a 38 percent solution commercially sold for stabilizing peroxide bleaching baths. The raw cotton utilized is run-of-the-mill gray cotton drawing sliver. The prebleached cotton is raw cotton which has been subjected to commercial kier-boiling and bleaching. The experimental ratios used were 2 to 1, 3 to 1, and 5 to 1 parts by weight of solution to cotton. The solution was prepared prior to each experiment and checked for its peroxide content. Weighed portions of cotton sliver were wet out with cooled bleaching solution, after which the excess of solution was squeezed out in order to bring the specimens to the desired weight. Duplicate sets were prepared for every experiment. Evolution data were secured from one set by means of a volumescope, and decomposition data were secured from the other set by titration. All experiments were conducted in a thermostatically controlled bath at $50° \pm 0.5°C$.

Figure 1 illustrates a volumescope, which consists of a test tube (1) fitted with a manometer made of capillary tubing (2) with a small bulb (3) blown at one end with a well (4) for indicator fluid sealed to the other end. A metallic scale (5) is attached along its length. The indicator fluid is a colored vaseline (6) which liquifies under the elevated temperature of the experiment. The manometer is fitted through a two-hole rubber stopper (7). Through the second hole of the stopper is placed a glass tube drawn out to a capillary tip (8) and the assembly attached to the test tube. The drawn capillary tip enables the system to be heat-sealed without compressing the gaseous contents.

The manometers were calibrated for use by injecting known volumes of air into the system under conditions simulating actual experiments [6] and observing the resultant displacement of the indicator fluid. During bleaching the units were immersed in a tall thermostat with the water level well above the top of the volumescope. At regular intervals readings were taken of the upper levels of the now liquid vaseline columns and converted into volume by means of a calibration chart.

The calibration and conversion of the observed scale readings are illustrated in Figure 2. Graph (a) depicts the calibration curve obtained for a volumescope when known volumes of air are injected into the system and the resultant displacement is observed in the manometer. This calibration was carried out for each volumescope.

During an experimental run, the scale readings at selected time intervals were plotted as in graph (b) and these data converted into volume by means of graph (a). Since the first 10–15 minutes are required to reach temperature equilibrium, the scale reading at zero time must be obtained by extrapolation. This is done by carrying out many observations in the first half hour, plotting these on an expanded coordinate graph system, and then extrapolating the resulting curve to zero minutes (see graph (c)). The volume represented by this zero reading is determined from graph (a) and thereafter subtracted from each observation to secure the corrected volume. The corrected volumes are then plotted against time as in graph (d).

The data on the decomposition of the bleaching solution

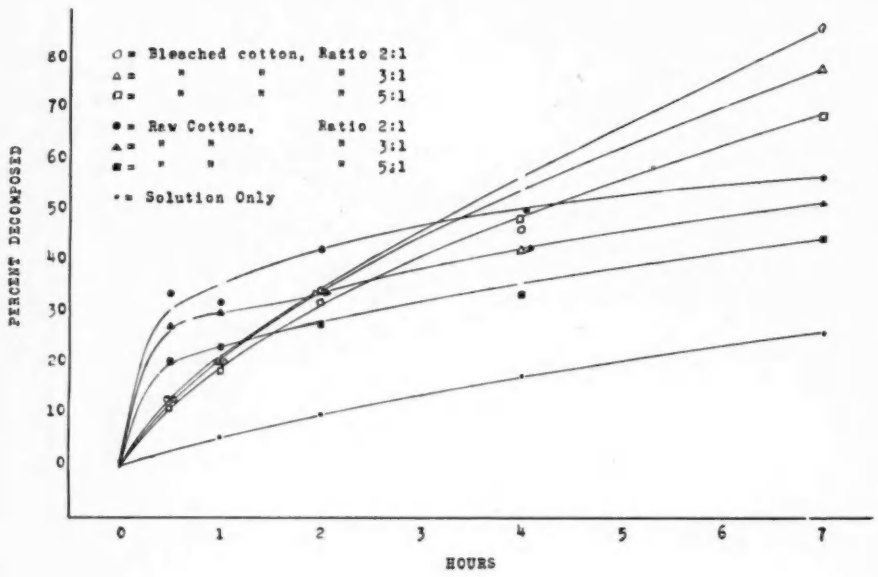

FIG. 3. *Decomposition of peroxide.*

were obtained from the second equivalent set. Each specimen was inserted into a test tube fitted with an air condenser and, with condenser emerging, was lowered into the thermostat. In each run, individual test tubes were withdrawn at the specified intervals. The reaction was quenched by cooling and flooding with diluted acid. The system was then diluted to a specified weight with water. These solutions were allowed to stand an hour with frequent shaking prior to withdrawing an aliquot portion for analysis by the standard iodimetric method for determining its remaining peroxide content [7]. Titration was carried out with a $N/100$ solution of sodium thiosulfate.

The decomposition rate of the bleaching solution in the absence of cotton was also determined.

The observed readings are calculated in terms of percent of initially available oxygen and graphed as functions of time. The data for plotting oxygen consumed are obtained as the difference between the plotted curves for the amount of decomposition and the amount of evolution.

Errors

The most serious potential sources of error as observed during the experiments are (1) air in fibers and solutions, (2) handling and preparation of specimens, (3) extrapolated oxygen evolution data, and (4) the fact that data for the decomposition and evolution were obtained from different specimens.

Calibration of the apparatus was effected under conditions simulating actual runs except that no hydrogen peroxide was used. This procedure was selected to compensate for air in the fibers, solution, or apparatus.

The error due to decomposition during sample preparation was eliminated by using a corrected value for T. This value was obtained by running a blank in which specimens were prepared in accordance with the usual procedure and analyzed immediately instead of being placed in the thermostatic bath.

The frequency of observations of the oxygen evolutions in the early stages of the bleach was increased and an expanded scale was used to minimize the errors inherent to extrapolation. All evolution data represent averages from duplicate runs. Repetition of the entire experimental procedure after an interval of six months gave satisfactory duplication of the original results.

Results and Discussion

Decomposition

Figure 3 illustrates the decomposition of the bleaching solution and the effect of the presence of bleached and unbleached cotton in varying ratios upon the decomposition of this solution. It is evident that the addition of cotton decreases the stability of the solution in the order of decreasing ratios. In the presence of raw cotton the amount decomposed is much greater in the early period than is observed for bleached cotton during the corresponding period. Thereafter the rate gradually slackens so that the total amount decomposed in the presence of bleached cotton eventually becomes greater than the decomposition due to the native cotton. Not only do the raw and bleached cotton differ in the total amount of oxygen decomposed but the characters of the curves themselves are entirely different. This indicates that

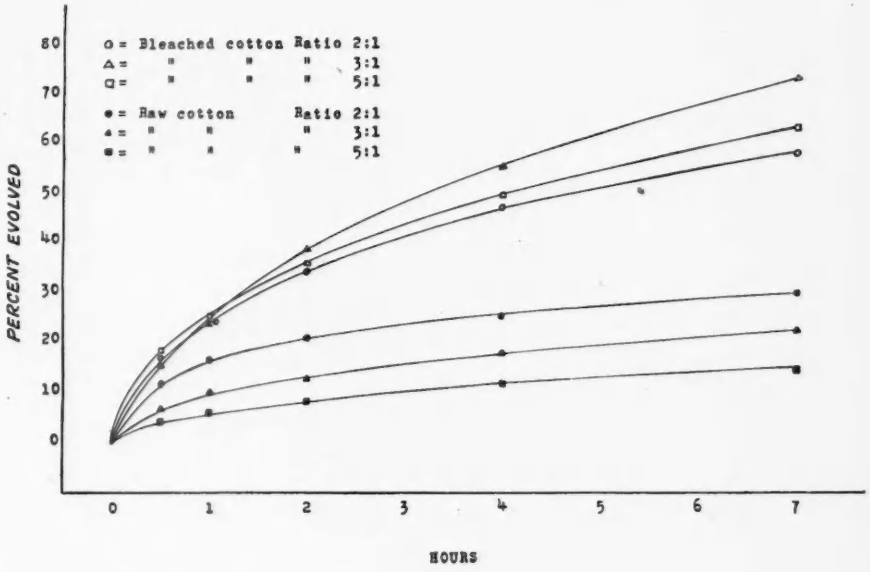

FIG. 4. *Evolution of oxygen.*

the decomposition in the presence of the bleached goods is of a distinctly different type from that occurring in the presence of raw cotton.

Oxygen Evolution

Figure 4 shows the percentage of the available oxygen evolved from the bleaching solution under the influence of bleached cotton and raw cotton. The amount evolved in the presence of prebleached cotton is much greater than that found when raw cotton is used. The influence of ratio upon this evolution follows a very orderly pattern in the presence of raw cotton; with the bleached specimens the 2:1 ratio appears misplaced. It is to be noted that in these curves the shapes are similar although the rates differ in magnitude. This is in contradistinction to what has been noted in Figure 3, where not only are the rates different for the two cottons but the shapes as well. This indicates that the mechanisms of the oxygen evolutions are fundamentally similar whereas the decompositions are not alike.

Oxygen Consumed

Figure 5 represents a series of curves depicting oxygen consumption calculated as the difference between the curves in Figures 3 and 4. This is in accord with equation (2). A striking difference between the reaction of raw and prebleached cotton is observable. As would be expected, the raw cotton consumed much more oxygen. The percent of oxygen consumed by the raw cotton is independent of the ratios in the range studied. The major portion of this consumption is effected in the first hour or two, after which the consumption proceeds at a much slower but steady rate. This speedy initial consumption accounts for the initial high decomposition rate of the bleaching solution observed in the presence of the raw cotton. On the other hand, some time elapses before the bleached cotton consumes any oxygen at all. Once initiated, however, the prebleached cotton consumes oxygen at a steady rate. In these consumption curves the difference in the behavior of the bleached and unbleached cotton bleaching systems is shown most clearly.

In the presence of prebleached cotton, the oxygen consumption is low, while the oxygen evolution rates and the peroxide decomposition rates are high. This low order of oxygen consumption coupled with the high peroxide decomposition rate in the presence of the prebleached cotton indicates a strong catalytic effect.

In the presence of raw cotton the high initial decomposition rate must be due to consumption by oxidizable material absent in the prebleached cotton. However, once this initial consumption has been effected, the character of the decomposition in the presence of the raw cotton is the same as that in the bleaching bath itself. This shows that after the reactive impurities of the raw cotton have been oxidized the solution behaves as though no cotton were present. This increased stability may occur because this experimental bleach either cannot create a catalytic surface on the raw cotton or, having produced a cata-

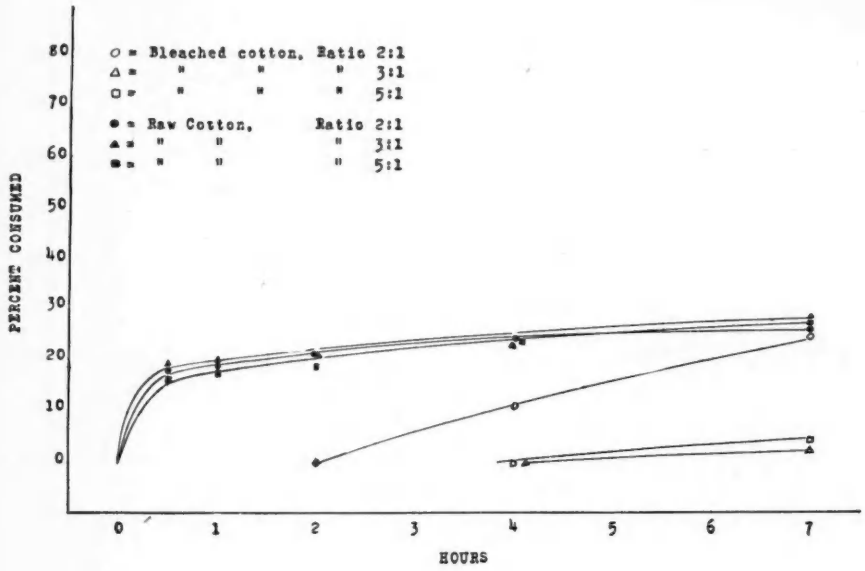

FIG. 5. *Consumption of oxygen.*

lytic surface, the reaction products of the oxygen consumption poison the surface. The dissolved reaction products may have a stabilizing influence. Whatever the mechanism, only the prebleached samples show undiminishing positive catalysis.

Conclusions

A satisfactory method has been developed for measuring the oxygen evolved during peroxide bleaching. These data in conjunction with those obtained from the decomposition of peroxide have provided a basis for quantitative estimation of the true oxygen consumption by the cotton. This will be useful for evaluating bleaching formulas. The interesting hypothesis developed during discussion of the data illustrates the worth in developing this procedure for investigating bleaching mechanisms.

Acknowledgment

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The Dyeometer: An Instrument for Studying Color Reactions

R. H. Kienle,* G. L. Royer,* and H. R. McCleary*

IN any extensive study of dyeing phenomena, it is desirable to be able to measure the rate at which dye is removed from the bath and transferred onto the material being dyed. In the past, the method of withdrawing portions of the dye bath during the course of dyeing and determining the color value photometrically has been most generally used. This procedure is cumbersome and not nearly so convenient as one which would permit the continuous observation of the dye bath. In order to make this possible an instrument called the Dyeometer was developed a number of years ago which has been improved since that time. This apparatus is mounted in a suitable metal support so that it can be moved into and out of the measuring instrument. The Dyeometer may be described as an apparatus which permits continuous photometric measurement of the strength of the dye bath during the course of the dyeing. It permits rigid control of every factor believed to influence the progress of dyeing; some of these factors are variation of temperature, volume of the dye bath, circulation of the dye bath, motion of the material being dyed, and control of the surrounding atmosphere. The Dyeometer has been found useful also in the study of any reaction in which a color change within a liquid phase is involved.

Other workers studying the dyeing process have determined the progress of exhaustion by periodic sampling of either the dye bath or the material being dyed [1]. Speakman and Smith [2] recognized the desirability of continuous observation and arranged for circulation of the dye bath through a Duboscq-type colorimeter, thus permitting measurements to be made at will. Davenport [3] has described an apparatus for circulating the dye bath through an observation chamber and reading the color strength by photoelectric means. Using this device, Lemin, Vickers, and Vickerstaff [4] have made some extensive measurements of exhaustion of direct dyes onto cotton. A considerable amount of data obtained with the apparatus described in the present paper has been published in several earlier papers from the Calco laboratories [5–10].

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Description of the Dyeometer

Constructional Details

Figures 1 and 2 are pictures of the apparatus showing the two sections, the dye-bath chamber and the cell unit, connected together by a ground-glass joint. A drawing of the dye-bath chamber is shown in Figure 3. The dimensions of the apparatus are such that 400 cc. of solution fills the dye-bath chamber to a level just below the side arm shown below the condenser section. A drawing of the Dyeometer cell unit is shown in Figure 4. It has been found useful to have several interchangeable cell units to permit the changing of the cell thickness at will and to have replacements immediately available if one cell unit is damaged.

The side view of the cell unit shows that the cell itself is offset from the ground-glass joint by about 1 inch. This has been done so that when the cell is placed in the measuring beam of the GE type of recording spectrophotometer it will be close to the integrating sphere. This type of recording spectrophotometer has been used to make most of the measurements reported here. It is desirable to have this cell as close to the integrating sphere as possible to avoid loss of light due to scattering. The drawing of the thermoregulator section (Figure 4) shows the bulb which is filled with mercury. The stopcock permits control of the temperature at any desired point simply by trapping various amounts of mercury in the thermoregulator bulb.

It is apparent that the Dyeometer can be used over a wide range of dye concentrations by having several cell units of different cell lengths. These cell units (made by the Scientific Glass Apparatus Company, Bloomfield, New Jersey) have been hand-blown with cell lengths as small as 1 to 1½ mm. They are not optically perfect but have been found to be sufficiently uniform in cell thickness across the face of the cell

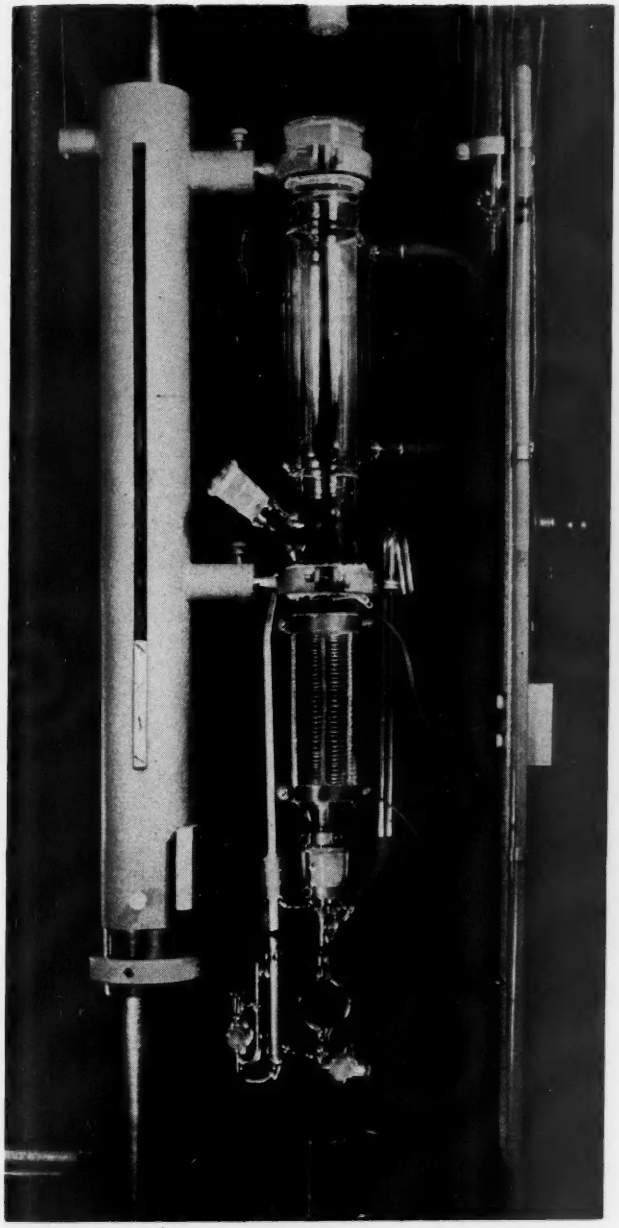

FIG. 1. *An over-all view of the Dycometer.*

for use in studying dyeings and similar color reactions. For cell thicknesses less than 1 to 1½ mm. a different type of cell has been designed. The drawings of such a cell are shown in Figures 5 and 6 (as a modification of Figure 4), and Figure 7 shows a picture of the entire cell unit. The cell thickness is determined by the thickness of the metal foil gasket which fits around the outside of the channel of the glass plate in Figure 6. This cell unit has been used with a 0.1-mm. lead foil in the study of concentrated dye solutions and appears to be quite satisfactory.

FIG. 2. *Close-up of the cell unit.*

Circulation and Stirring

Circulation of the dye bath is achieved by the use of a gas lift. The dye-bath liquor circulates from the cell unit up through the center of the stirrer or circulating tube (Figures 8 and 9) and flows out through the hole at the top into the main dye bath. This provides continuous circulation of the bath past the thermoregulator and through the observation cell. The time required for complete circulation of the 400-cc. bath is about 7 to 12 seconds. Nitrogen is usually used for the gas lift because it is inert and can therefore be used with vat dyes.

Figure 10 shows a drawing of the glass cap which fits into the top female joint of the large dye-bath section; the hole in the center accommodates the stirrers while the side arm tube leads to an auxiliary condenser to prevent evaporation of the bath. An auxiliary condenser was found to be necessary because the main condenser at the top of the dye-bath section is not adequate.

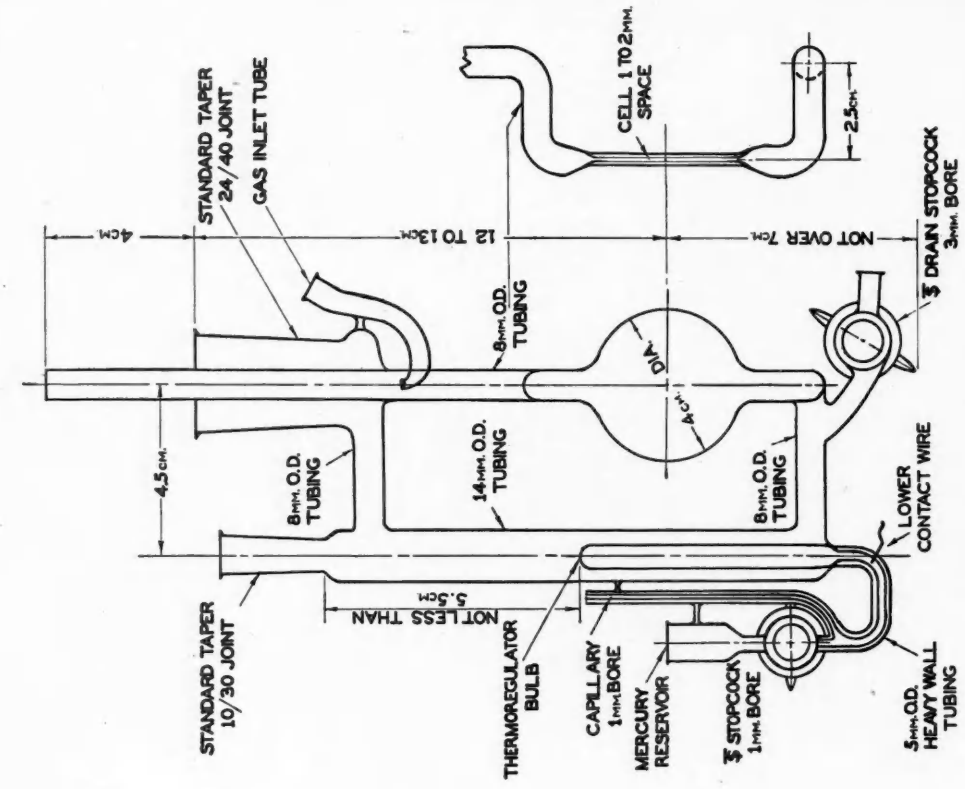

FIG. 4. *Drawing of the cell unit.*

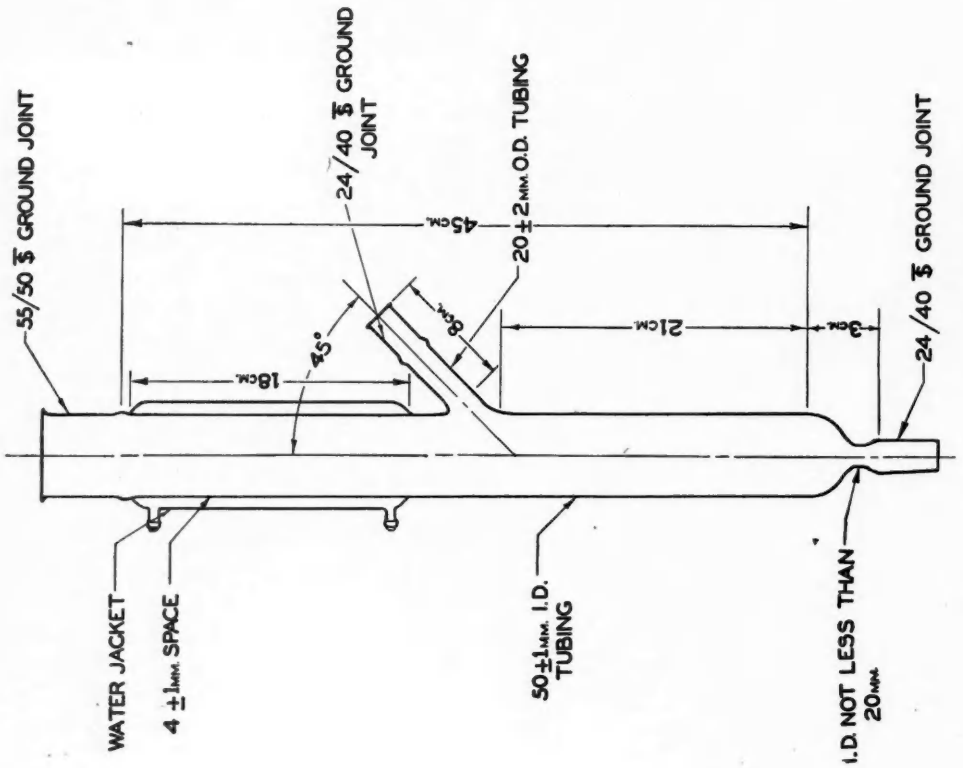

FIG. 3. *Drawing of the dye-bath chamber.*

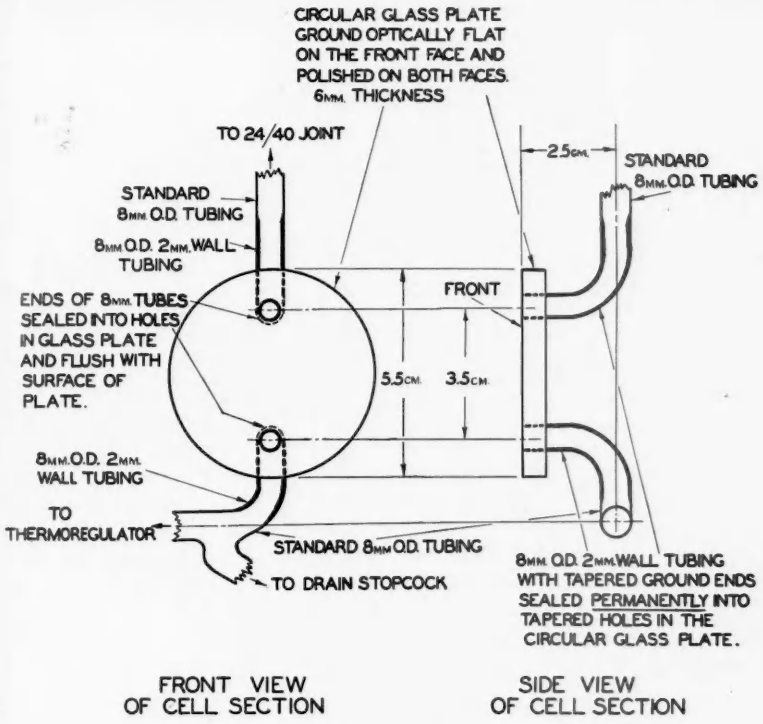
CIRCULAR GLASS PLATE GROUND OPTICALLY FLAT ON THE FRONT FACE AND POLISHED ON BOTH FACES. 6MM. THICKNESS
TO 24/40 JOINT
2.5CM.
STANDARD 8MM.O.D. TUBING
STANDARD 8MM O.D. TUBING
8MM O.D. 2MM. WALL TUBING
FRONT
ENDS OF 8MM. TUBES SEALED INTO HOLES IN GLASS PLATE AND FLUSH WITH SURFACE OF PLATE.
5.5CM.
3.5CM.
8MM.O.D. 2MM. WALL TUBING
TO THERMOREGULATOR
STANDARD 8MM O.D. TUBING
TO DRAIN STOPCOCK
8MM. O.D. 2MM. WALL TUBING WITH TAPERED GROUND ENDS SEALED PERMANENTLY INTO TAPERED HOLES IN THE CIRCULAR GLASS PLATE.
FRONT VIEW OF CELL SECTION
SIDE VIEW OF CELL SECTION

FIG. 5. *Drawing of the cell of the thin cell unit.*

In carrying out skein or cloth dyeings the material to be dyed is fastened onto a stirrer of the type shown in "B" of Figure 8. It may be noted that this is identical with the circulating tube except that small glass hooks have been sealed onto the outer portion of the tube to which the skein may be attached. The top hooks have small eyes through the ends to permit tying a small string or thread around the top to prevent the material from coming off the hooks. This stirrer has been found suitable also for carrying out dyeings on woven cloth by cutting small holes in the cloth and fastening them onto these hooks. A type of basket stirrer such as has been used for the dyeing of raw stock materials is shown in Figure 9. This again is like the circulating tube except that a basket or cage has been attached to the outer portion of this tube by fusing 3-mm. glass rod to the tube in the form of a cage. The four vertical edges of the cage have been indented so that string may be tied around the cage to prevent small portions of raw stock from coming loose during the course of the dyeing in the bath.

In use, one of the stirrers is suspended in the main body of the dye bath through the hole in the dye-bath cap and attached above the cap to a suitable motor. The motion of the stirrer is back-and-forth through an angle of about 270 degrees. In determining the rate of oscillating of the stirrer required for reproducible dyeings, the "cage" stirrer for raw stock has been used because rate of stirring is most critical in this type of dyeing. Figure 11 shows a plot of percent dye-bath strength against time for three dyeings of Calcomine Sky Blue FF (C.I. 518) on raw stock cotton at various stirring rates; one complete back-and-forth motion of the fiber holder is one cycle. It may be seen that the rate of exhaustion increases with increase in stirring rate up to 50 cycles per minute. Higher rates of stirring show the same exhaustion rate. Therefore, the synchronous motor which is used to drive the gear mechanism for the stirring has been chosen so that the stirrer operates at a speed of 60 cycles per minute. A large number of dyeings shows that this rate of stirring is sufficient to provide reproducible results in practically all dyeings which may be carried out on the Dyeometer.

In the measurement of very fast reactions such as the reduction of vat dyes at higher temperatures, the rate of circulation of the dye-bath liquor through the observation cell is of critical importance; that is to say, reactions or changes which may take place in the main body of the dye-bath chamber must be

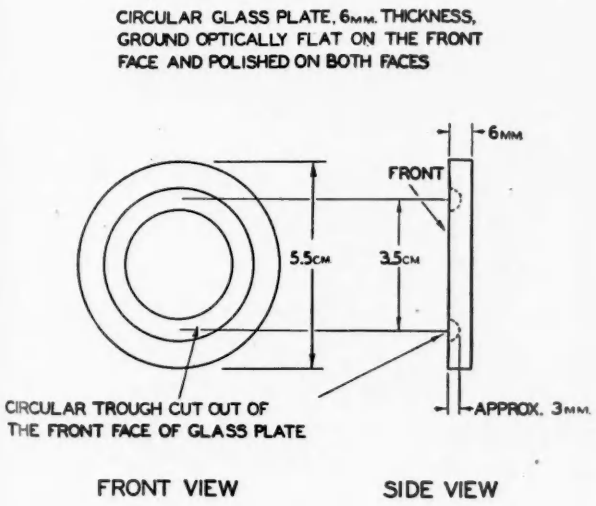
CIRCULAR GLASS PLATE, 6MM. THICKNESS, GROUND OPTICALLY FLAT ON THE FRONT FACE AND POLISHED ON BOTH FACES
6MM
FRONT
5.5CM
3.5CM
CIRCULAR TROUGH CUT OUT OF THE FRONT FACE OF GLASS PLATE
APPROX. 3MM.
FRONT VIEW
SIDE VIEW

FIG. 6. *Drawing of channeled glass plate for thin cell unit.*

accompanied by identical changes in the observation cell within a very short period of time. This time, designated as the mixing time, determines the usefulness of this apparatus in measuring very rapid reactions. The mixing time has been determined by placing the Dyeometer in the photometric apparatus, filling the Dyeometer with a water solution only, and adjusting the nitrogen flow to provide optimum mixing and then adding a few cubic centimeters of a very strong dye solution. With the spectrophotometer turned on it is thus possible to determine the maximum amount of time for the pen of the recording mechanism to come to equilibrium, at which point the bath may be considered to be completely mixed. With the nitrogen flow set at what appears to be optimum mixing, the bath requires about 12 seconds to mix completely. The rate of nitrogen flow appears actually to be of little importance in determining the efficiency of mixing as long as it is high enough to provide continuous circulation of the dye bath. In the mixing experiment just described, it was observed that there was some stratification of the dye solution which was added; that is to say, a very strong solution passed through the cell, then a weaker solution, then a stronger, then a weaker, etc., until the bath became thoroughly mixed. In order to eliminate this, the mixing experiment was repeated by inserting a long glass tube into the top of the dye-bath portion and passing through this tube a stream of nitrogen from another tank. This auxiliary tube causes rapid mixing of the main body of the dye-bath and the original circulation achieved by the first stream of nitrogen circulates this main dye bath through the cell section. By this procedure, it was found that the mixing time is of the order of 7–10 seconds.

Heating and Temperature Control of Bath

The bath is heated electrically by No. 22 nichrome wire (total resistance of 20 ohms) wound around the main body of the dye-bath chamber. The voltage applied to the heater is determined by the thermoregulator. Figure 12 shows the detailed wiring diagram of the Dyeometer control panel, a picture of which is shown in Figure 13. The method for controlling the temperature may be stated briefly as follows: The current flows to the nichrome-wire heating coil from either of two variable transformers depending upon the position of the thermoregulator. Thus, if the iron regulator wire is touching the mercury surface in the capillary tube of the thermoregulator, then the current is flowing from the "low heat" variable transformer. If, on the other hand, the regulator wire is not touching the mercury in the thermoregulator capillary tube, then the current is flowing from the "high heat" variable transformer. The voltages on the "high heat" and the "low heat" transformers are chosen at will to give optimum temperature control and depend upon the temperature which it is desired to maintain. When the wire in the thermoregulator capillary touches the mercury surface, it trips an electronic relay (Fisher-Serfass Electronic Relay, Fisher Scientific Company), which in turn trips a heavier, 6-volt relay; this relay connects one or the other transformer to the heating coil.

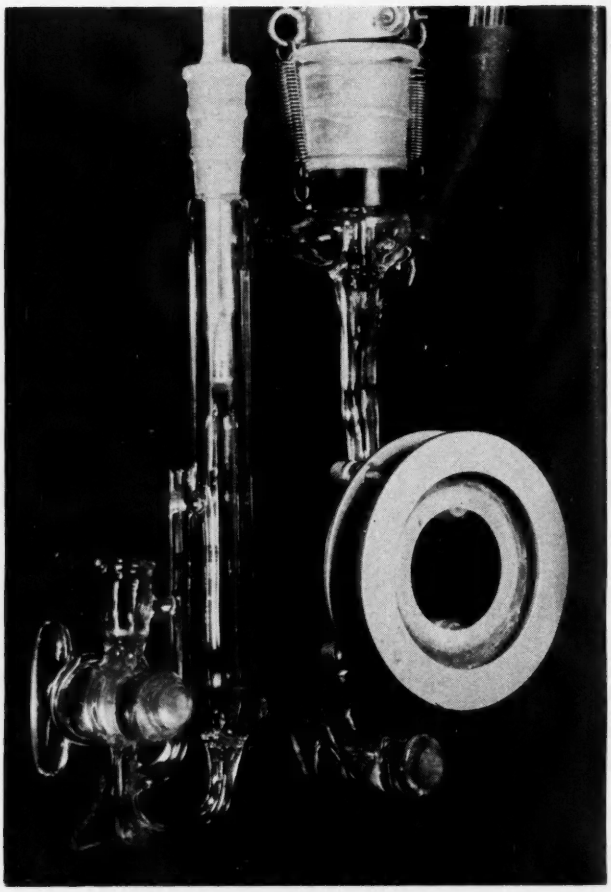

FIG. 7. *Close-up of thin cell unit.*

Uses of the Dyeometer

Use of the Dyeometer in Dyeing

The following procedure is the method followed in carrying out a dyeing in the Dyeometer: A 400-cc.

dye bath is prepared. The nitrogen is turned on and the bath is poured into the Dyeometer. The circulating tube is inserted into the top of the Dyeometer and fitted over the extension of the cell unit tube. This circulating tube is supported by a two-hole rubber stopper which contains a side arm tube leading to the auxiliary condenser. The main switch and the "low heat" and "high heat" switches are turned on. The regulator wire is adjusted so that it does not touch the mercury surface. The indicator light will turn on to show that the bath is heating. For example, if the dyeing is to be carried out at 90°C (195°F), the "high heat" transformer is set at a voltage of about 70–80 volts to be adjusted later for accurate temperature regulation. This voltage setting varies considerably depending upon the temperature of the room and the rate of air flow in the room itself, both of which affect the rate of heat loss from the Dyeometer. When the thermometer indicates that the bath has reached a temperature of 90°C, the thermoregulator stopcock is then closed and the regulator wire is inserted into the capillary until it is just touching the mercury, thus shutting off the indicator light. At this point it is essential to set the two variable transformers correctly. The "high heat" transformer should be set at a voltage which is just enough to make the bath heat slowly when the indicator light shows that it is heating, while the "low heat" transformer should be set at a voltage just below that required to hold the desired temperature when the indicator light is off, showing that the bath is cooling. Thus, the expansion and contraction of the mercury and liquid in the thermoregulator makes and breaks contact with the regulator wire in the capillary and chooses between a condition where the bath is slowly cooling and one in which it is slowly heating. This condition is optimum for maintaining accurate temperature control and if these controls are properly set, one may maintain the temperature of the Dye-

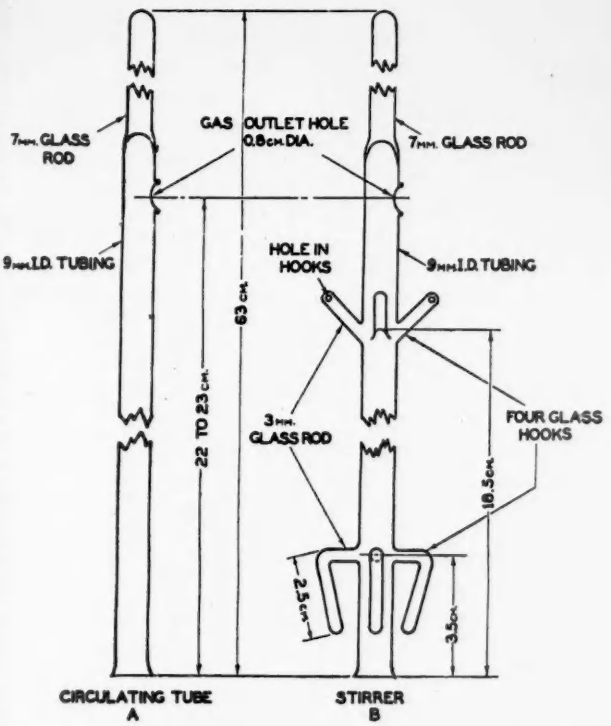

FIG. 8. *Drawings of circulating tube and skein holder stirrer.*

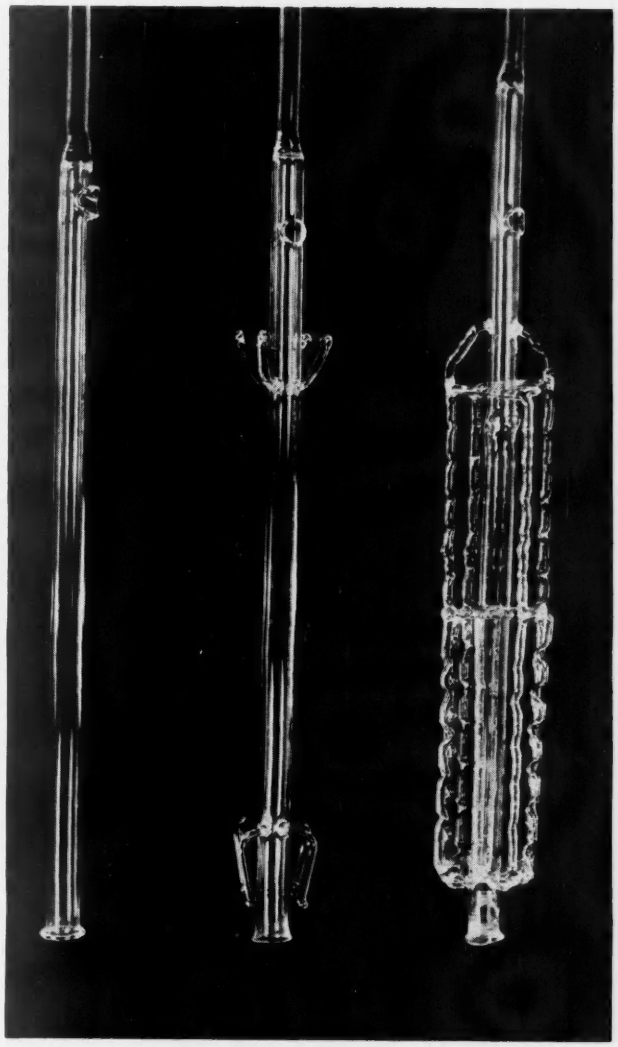

FIG. 9. *View of circulating tube and stirrer holders for skeins and raw stock.*

ometer with adequate circulation to within ± 0.2° to 0.3°C.

It may be mentioned here that the Fisher-Serfass Electronic Relay used in the Dyeometer heat-control unit is exceedingly sensitive; the two wires leading to the regulator wire and the mercury contact of the thermoregulator will trip the relay when shorted through a resistance as high as 20 megohms. For this reason many common liquids, including tap water, have sufficient conductivity to trip the relay. If, therefore, water should be spilled into the thermoregulator capillary, the relay will be tripped even though the regulator wire is not touching the mercury surface.

While the bath is heating, the skein or other material to be dyed is prepared and attached to the appropriate stirrer. If skeins are to be dyed, it has been found that not more than 15 grams of material may be dyed conveniently in the dye bath. Twenty grams or more packs the dye bath so tightly that the stirring is inadequate and the material is unevenly dyed. Optimum dyeing results are obtained if only 5 or 10 grams of material are used. Skeins are fastened loosely on the stirrer rather than being pulled tightly over the hooks because if pulled tightly they will not dye uniformly.

When the bath has reached the dyeing temperature, a photometric reading is made on this initial bath. The dyeing is started by entering the stirrer containing the material into the bath, and the stirrer is attached to the stirring mechanism above the Dyeometer and the motor is started. At specific time intervals, photometric measurements of the bath strength are determined. These measurements may be made with a suitable recording colorimeter [11] or by use of a spectrophotometer [12]. As described by Stearns [8], the complete spectrophotometric curve may be run at stated intervals if the exhaustion is slow. If, however, exhaustion is rapid, only a small segment of the spectrophotometric curve, usually at the absorption maximum, may be run. For very rapid exhaustions one may set the wave length at the absorption maximum and plot time as the abscissa against transmittance as the ordinate. This method is less informative because it does not give the spectrophotometric curve showing possible color changes, but is valuable if this limitation is recognized. When the dyeing has been carried out for the desired length of time, the heater switch is turned off and the dye bath is drained.

In reading the data, one must correct for the transmittance of the Dyeometer cell filled only with the water solution without the dye. It is found most suitable to run a spectrophotometric curve of the water-filled Dyeometer cell and to use this curve as the correction curve for all of the curves obtained during the dyeing. If some agent or assistant has been added to the dye bath which may also cause some light absorption, then the correction should be made with a water solution containing this agent or assistant. Most dyeing assistants or similar agents such as inorganic salts, acids, etc., are colorless and do not contribute to the absorption of the water-filled cell. The corrected transmittance values are then used to calculate the percentage dye-bath strength in terms of the original dye-bath concentration taken as 100 percent. A dye-bath exhaustion curve is then drawn on a graph of percent dye-bath strength as the ordinate against time as the abscissa. Typical curves of this type are shown in Figure 11.

The Dyeometer has been found useful in a scientific study of the dyeing of fibers such as cotton, rayon,

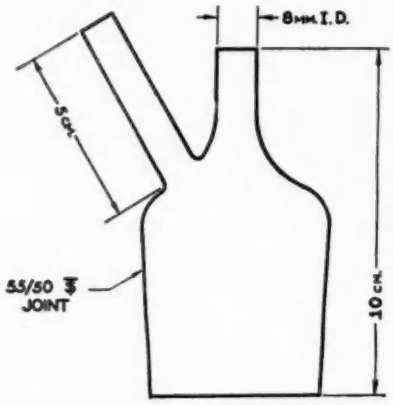

FIG. 10. *Drawing of cap for dye-bath chamber.*

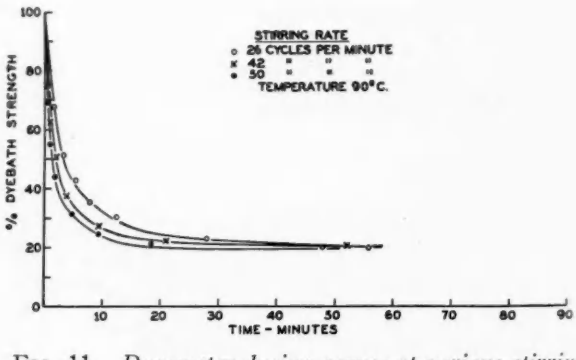

FIG. 11. *Dyeometer dyeing curves at various stirring rates; dyeing temperature 90° C, 5-gram cotton sample, 400-cc. bath containing 0.100 gram Calcomine Sky Blue FF Ex. Conc. and 5 grams NaCl.*

casein fibers, wool, nylon, etc. Dyeing of these fibers has been investigated from two points of view, namely, variation of the dye with the same fiber and variation of the fiber using one dye. For example, a previous paper [9] has described the effect of various pretreating and finishing treatments on the dyeing of wool. Similar experiments have been carried out on the other textile fibers mentioned above. Figure 14 shows a set of exhaustion curves for dyeings of Calcomine Sky Blue FF on rayon of various filament sizes manufactured from the same batch of viscose.* It is proposed to discuss the use of the Dyeometer in the dyeing of different types of fibers more completely in subsequent papers.

Comparison of Dyeometer and Mill Dyeing Data

In cooperation with several commercial dyers of wool, a study has been made to compare the data from mill dyeings with those of Dyeometer dyeings using the same dyeing formula. It was found in all cases that the rate of exhaustion of the bath in the Dyeometer was somewhat faster than in the corresponding mill dyeing. It is believed that this difference may be due to the more efficient circulation of the dye liquor through the material in the Dyeometer. Similarly, it is known that laboratory beaker dyeings cannot be compared directly with mill dyeings. However, as in the case of beaker dyeings, the Dyeometer dyeings can be compared with each other and used to predict the results of mill dyeings when proper corrections are applied.

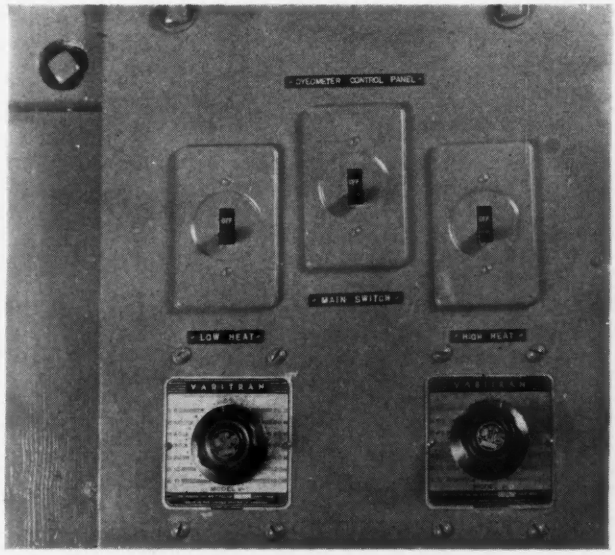

FIG. 13. *View of Dyeometer control panel.*

Use of the Dyeometer in the Study of Vat Reduction Rates

The Dyeometer may be used not only for following the course of a dyeing, but may be used also in following the rate of other color reactions—for example, the reduction of vat dyes. A study of the rate of reduction of a vat dye involves the determination of how fast the insoluble vat dye dispersion is changed to the soluble leuco form. The rate determined for a given sample of dye is affected by temperature, concentration of alkali and hydrosulfite, and by rate of mixing and circulation of the bath through the Dyeometer cell. A typical procedure for determining reduction rate of a vat dye is as follows: A 400-cc. bath is prepared containing 0.1 to 0.6 gram of any dispersed vat-dye paste and 25 ml. of 7.5 *M* sodium hydroxide (equivalent to 2½ oz. per gal.). This bath is placed in the Dyeometer, heated to 140°F (60°C), and the thermoregulator is set at this temperature.

* These samples were obtained through the courtesy of the Research Laboratories of the American Viscose Corporation, Marcus Hook, Pennsylvania.

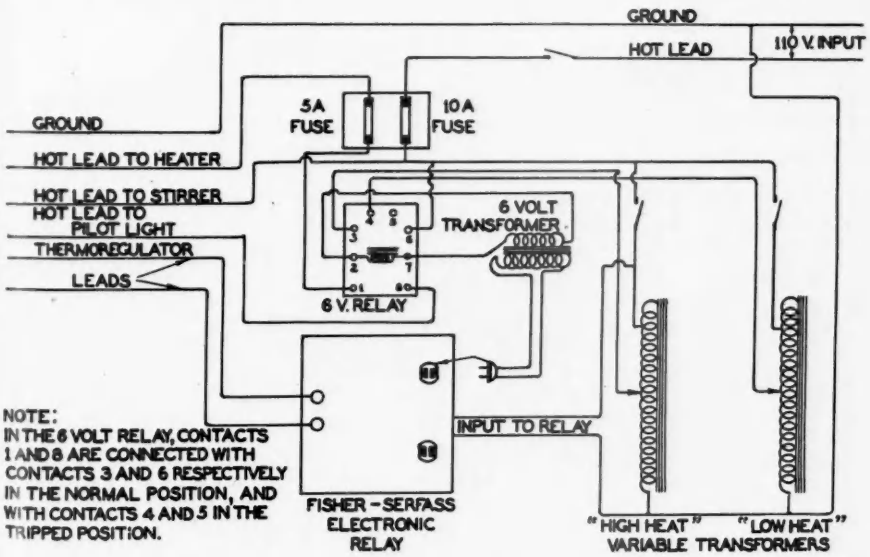
GROUND
HOT LEAD
110 V. INPUT
5A FUSE
10A FUSE
GROUND
HOT LEAD TO HEATER
HOT LEAD TO STIRRER
HOT LEAD TO PILOT LIGHT
THERMOREGULATOR
LEADS
6 VOLT TRANSFORMER
6 V. RELAY
NOTE:
IN THE 6 VOLT RELAY, CONTACTS 1 AND 8 ARE CONNECTED WITH CONTACTS 3 AND 6 RESPECTIVELY IN THE NORMAL POSITION, AND WITH CONTACTS 4 AND 5 IN THE TRIPPED POSITION.
INPUT TO RELAY
FISHER-SERFASS ELECTRONIC RELAY
"HIGH HEAT" VARIABLE TRANSFORMERS
"LOW HEAT"

FIG. 12. *Wiring diagram for Dyeometer control panel.*

TABLE I.

Dye	% Reduction at 10 sec.	15 sec.	20 sec.	Time (sec.) required for 97–100% reduction
Calcoloid Black BBD Double Powder	100			10 or less
Calcoloid Gray RD Paste	100			10 or less
Calcosol Blue GCD Double Paste	82	90	95	25 to 30
Calcosol Dark Blue BO Paste	95			15 to 20
Calcosol Blue R Double Paste	97			10 to 20
Calcosol Brown R Paste	90	94	96	25 to 30
Calcosol Jade Green N Double Paste	100			10
Calcosol Blue Green FFBP Double Paste	80	90	95	25 to 30
Calcosol Olive Green BN Paste	100			10 or less
Calcoloid Golden Orange GD Double Powder	90	93	96	25 to 30
Calcosol Pink FFD Paste	33	47	62	>120 (unstable)
Calcosol Violet RR Extra Paste	100			10 or less
Calcosol Violet 6RD Paste	65	80	85	60 to 80
Calcoloid Yellow GCD Double Powder	95			20 to 25
Calcosol Yellow PR Double Paste	83	92	95	30 to 40

The spectrophotometer is set at the wave length which shows the greatest change of transmittance on reduction of the dye to the leuco form, the spectrophotometric curve of which has previously been determined. Three grams of sodium hydrosulfite (equivalent to 1 oz. per gal.) are added and the change of transmission is followed on the spectrophotometer. Since most vat reductions take place quite rapidly, the procedure described above for following rapid dyeings is used. From the data thus obtained the rate of formation of the leuco form may be calculated. This calculation from spectrophotometric data assumes that the spectral curve of the insoluble vat dispersion is that of a true colored solution; although this is not strictly true, the results are sufficiently reliable to permit comparisons of reduction rates of various dyes to be made under various conditions. Reduction curves obtained in this manner have been published by Clark [10]. Figure 15 shows typical reduction curves for Calcoloid Yellow GCD at three different temperatures; the marked effect of temperature on the reduction rate is quite evident.

Recent developments in the continuous dyeing of vat colors have led to their application at temperatures as high as 200°F. This indicates the necessity for using dyes which reduce rapidly under these conditions. The Dyeometer has proved useful in determining the time required for reduction at this temperature in known concentrations of caustic and hydro. Table I lists some vat dyes with data on their rates of reduction at 200°F. The dyes listed were chosen from the slower-reducing types in order to show the measurements which can be made on the Dyeometer.

In these determinations the mixing time of the

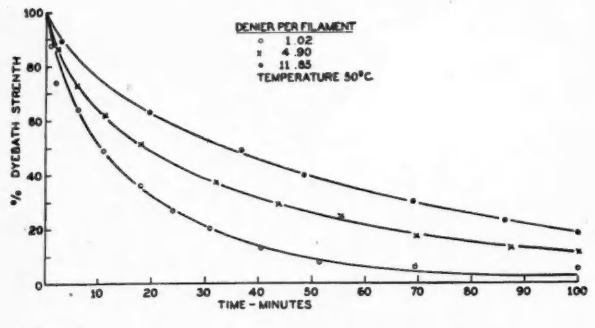

FIG. 14. *Dyeometer dyeing curves of rayons of various filament sizes; dyeing temperature 50° C, 10-gram rayon skein, 400-cc. bath containing 0.200 gram Calcomine Sky Blue FF Ex. Conc. and 1 gram NaCl.*

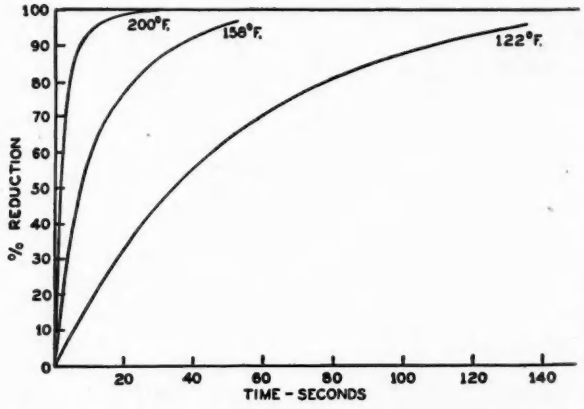

FIG. 15. *Reduction curves for Calcoloid Yellow GCD Dbl. Pdr. reductions in the Dyeometer at various temperatures.*

bath in the Dyeometer was 7–10 seconds, as discussed in the section "Circulation and Stirring." Therefore, no values are given in the table for reduction times of less than 10 seconds. It is felt that the publication of these data may be of value to those who are interested in the use of vat dyes under these conditions of application.

Use of Dyeometer in Determination of Rates of Chromation

In the dyeing of wool with the so-called chrome dyes the rate at which the chrome-dye complex is formed in the dye bath during the dyeing operation is important. This is true because frequently the chrome-dye complex is not as readily absorbed by the wool as the unchromed dye. Therefore, the strength and shade of such a dyeing is affected by the rate of chromation. The chrome-dye complexes on the fiber are usually faster to light and washing than the corresponding unchromed dyes; it is therefore important that the dye be applied in such a way that it will be completely chromed in the finished dyeing. A study of the rates of chromation of dyes in solution yields considerable information on the comparative properties of the chrome dyes.

The Dyeometer is well suited for making measurements on rates of chromation of dyes. Such experiments can be carried out by making spectrophotometric records on the dye bath during a dyeing or during a chromation in the absence of any fiber. The effect of variations in temperature, bath composition, and other factors on both chromation and dyeing can be studied. A typical family of curves for the chromation at the boil of Calcochrome Fast Red ECB (C.I. 652) with potassium dichromate in the absence of wool at a pH of 5 to 6 is shown in Figure 16. Curve A was taken just before the addition of potassium dichromate and represents the color of the unchromed dye. Curve F represents the color of the chromed dye formed at the end of 30 minutes; there was no further change in the curve up to an hour and a half. Intermediate curves represent the color of the bath during the chromation. From these data one may calculate the degree of chromation at various times; a plot of these data is shown in Figure 17.

An examination of the curves in Figure 16 shows that they have a common crossing point at about 515 mμ. This indicates that during the chromation reaction there is no appreciable change in concentration of dye. If a dyeing is made using these same conditions, it is possible to make measurements at this wave length and determine the extent of total exhaustion. The amounts of chromed and unchromed dye remaining in the dye bath at any time could then be determined by an analysis of the respective curves.

Other Uses for the Dyeometer

The Dyeometer is so constructed that experiments can be carried out under a variety of controlled conditions. Thus, one may measure the stability of

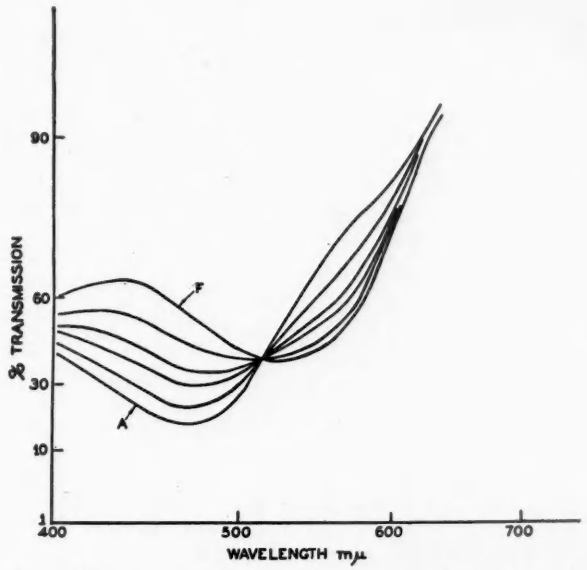

FIG. 16. *Spectrophotometric curves obtained during the chromation of Calcochrome Fast Red ECB at 93° C and a pH of 5.7 following addition of $K_2Cr_2O_7$ to the bath.*

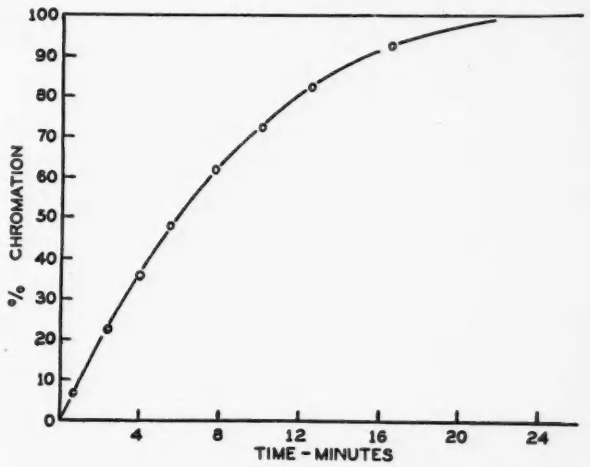

FIG. 17. *Plot of percent of dye which has been chromed at various times; calculated from curves of Figure 16.*

dye baths or colored solutions under an atmosphere of nitrogen to eliminate any possibility of air oxidation. One might also use oxygen, chlorine, or any other gas to achieve circulation in place of the nitrogen in order to study any color reaction brought about by a saturated solution of the given gas. The stability of dye solutions can be determined at any desired temperature and for any length of time. In this connection, a particular advantage of the Dyeometer is that the spectrophotometric record obtained represents the color of the dye solution at that particular temperature and under the exact conditions of experiment; this would not be true if aliquot samples were withdrawn for measurement.

It is obvious that the Dyeometer has many other applications in the study of the formation of colored substances such as pigments, dyes, and metallic complexes; and it can also be used in carrying out titrations in which color reactions are involved. In fact, the Dyeometer may be used to follow any reaction taking place in a liquid system where spectrophotometric changes occur during the course of the reaction.

Acknowledgment

The authors of this paper wish to acknowledge the help of their many associates who have worked from time to time either on the development of this instrument or the obtaining of data. Special credit is due to Mr. C. H. Benbrook, who did much of the early work in the design and use of the first Dyeometers, and to Dr. M. L. Crossley for his continued support of this project.

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Solvent Extraction of Cotton Wax

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Solvent extractions of many samples of cotton showed that the usual methods of wax extraction are not always adequate for general use, particularly on cottons whose extractable matter has been changed by manufacturing operations such as the addition of spinning oil or warp size, or the removal of part of the wax by bleaching processes. The usual simple extractions may be satisfactory for routine work on raw or processed cottons if the solvent is selected according to the type of cotton and to the purpose of the extraction. If, however, an accurate estimation of amount of wax is needed, a more inclusive method is desirable. The alcohol-chloroform extraction procedure developed by Conrad is shown to be such a method. Several of the various other methods of extraction discussed are more suitable for dewaxing cotton for special uses than for analysis for wax content.

Introduction

The analytical values obtained from the solvent extraction of various samples of unbleached, partially bleached, and bleached cottons in the course of a broad program of research on cotton bleaching have been higher than expected from data in the literature on bleaching and higher than the values usually obtained by conventional methods of extraction.

The crude material, usually called cotton wax, extracted from cotton by chloroform, carbon tetrachloride, benzene, or similar organic solvents, though present in comparatively small quantities, plays an important part in the manufacturing and finishing of cotton yarns and fabrics. The amount of wax present influences the classer's estimate of fibers [1] and has been said [29] to be the chief cause of discolorations of bleached fabrics during storage. Hess has been credited with the statement [30] that we cannot understand the chemistry of cellulose itself without a knowledge of the materials that accompany it. In the present program of research, the amount of residual wax has proved to be one of the most useful indications of the effectiveness of kier-boiling and bleaching processes.

The object of this report, therefore, is to present usable data on the extraction of wax from identical cottons by means of a number of different solvents under the same laboratory conditions, techniques, and methods which are adaptable to analytical procedures.

Literature Review

Information on cotton wax and its extraction from fibers, yarns, and fabrics is both limited and widely scattered. Much of it has appeared in scientific journals which are not readily available. When found, these data are confusing and not comparable. The commonly used methods for the dewaxing of cotton for analytical or research purposes utilize periods of extraction varying from 4 hours [1, 2, 4] to two successive periods of 24 hours each [27] and use a great variety of solvents, techniques, and temperatures which are frequently inadequately specified.

Benzene [13, 22], chloroform [6, 12], carbon tetrachloride [20], ethyl alcohol [9], isopropyl alcohol [17], alcohol-benzene mixture [15], trichloroethylene [10], and ether and other solvents [5] have been used or recommended for extraction of the cotton wax. Hess [14] credits Schwalbe with recommending equal parts of ethyl alcohol and benzene. Hess also mentions extraction by ether followed by ethyl alcohol as effective and states that chloroform and benzol are especially good as solvents for both resins and waxes, whereas carbon tetrachloride dissolves only the resins. Sookne and Harris [27] extracted cotton for 24 hours at room temperature with 95-percent ethyl alcohol, followed by 24 hours' extraction with diethyl ether, again at room temperature. They also extracted with hot ethyl alcohol

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for 24 hours and then washed the cotton with distilled water at room temperature for several hours [25, 26]. In both cases, the product was called "dewaxed cotton."

Bloor [3] and Maclean and Maclean [21] have recognized that hot ethyl alcohol is probably the best solvent for extraction of fatty material from biological tissues. Bloor [3] is emphatic about the superiority of boiling alcohol to all other solvents for the purpose. He states that it can be applied directly to the tissues without previous drying, and the almost inevitable accompanying oxidation, breaking up whatever loose combinations there may be between the lipide and other cellular constituent. Maclean and Maclean state [21]: "On drying . . . and extracting . . . with ether, no lipin whatever was obtained while subsequent treatment with alcohol removed a considerable amount."

Bloor mentioned two disadvantages of the extraction with hot ethyl alcohol: Substances other than lipides are extracted, and heat decomposes some of the more sensitive lipides. The first of these disadvantages is largely eliminated, however, with use of Conrad techniques [9, 17] since this analytical procedure rids the extract of all nonwax materials. The second disadvantage hardly exists since it is probably true with raw cotton, and certainly true with processed cotton, that sensitive lipides have already been decomposed before sampling.

The statement by Tonn and Schoch [28] that diethyl ether, benzene, toluene, Skelly solvents, chloroform, and carbon tetrachloride "gave complete removal of the wax from the fiber" should have been qualified. While extractions with these solvents may have been adequate for the purposes Tonn and Schoch had in mind, their statement does not agree with the experience of the Southern Regional Research Laboratory, or with the findings of Clifford, Higginbotham, and Fargher [5]. Moreover, Knecht and Hall [18] found that alcohol following benzene extracted an additional quantity from both Egyptian and American cottons.

Ahmad and Sen [1] used a 4-hour period for the extraction of cotton wax. The same period is specified by A.S.T.M. [2] and by the British Standards [4] for the following solvents: ether, methylene chloride, alcohol, and benzene-methyl alcohol mixture. The U. S. Pharmacopoeia [23, p. 207] specifies a 5-hour extraction. These methods were designed mainly for control and routine specification. For comparative purposes, Clifford, Higginbotham, and Fargher [5] suggest 6 hours, and Conrad [9] specifies 6 hours, and hot extraction (above 55°C).

Since Conrad [9] and Tonn and Schoch [28] have recently reviewed the literature pertaining to the complex, heterogeneous nature of cotton wax and its extraction from the fiber, further discussion of the literature in this report is unnecessary.

Materials and Sampling

In a series of preliminary experiments run for the purpose of determining the time of extraction, the material used was an Empire cotton, classed as "strict low middling, bright; character slightly irregular"; it was cleaned, carded, drawn once, and stored as sliver lap until used. This material was more uniform than the average cotton lint and was not, therefore, given any further mixing to insure uniformity. After conditioning 4 hours, approximately 16-gram samples were weighed accurately on an analytical balance. Triplicate moisture samples were weighed at the same time.

For a second series of experiments, designed to determine the degradation of cotton caused by extended extraction, four pieces of print cloth, two of which were slightly degraded, were chosen because they contained no blemishes or irregularities. Accurately weighed 10-gram samples were extracted without previous systematic or uniform sampling.

A third series of experiments was run to determine the comparative effectiveness of a number of solvents when used under the same laboratory conditions and techniques. The materials used were the Empire raw cotton already described, a cotton mesh bagging material which had been kier-boiled and subsequently soap-scoured, and five pieces of broadcloth, each having had a different purification treatment. The samples, which do not represent successive stages in the same process but are the residual material after different types of processing, were selected because their known wax content and scouring or bleaching treatment made them suitable for the purpose. The wax contents shown in Table III should not be taken as representative of the treatments used in the preparation of the materials. Very little distinction has so far been made between extractions of cotton wax, wax plus the fatty ingredients of textile sizes, or residual wax which has survived such treatments as kier-boiling and bleaching. The foregoing samples were so selected that they represent these types of materials as well as raw cotton.

To insure uniformity of the material in this third series of experiments each sample was cut into small pieces, the pieces thoroughly mixed, and then put through a No. 1 Wiley mill to pass a 2-millimeter screen. Each ground sample was rolled, quartered, mixed on a large piece of wrapping paper, and again put through the Wiley mill. The rolling, mixing, and grinding were repeated so that each sample passed three times through the Wiley mill and 2-millimeter screen.

Air-dried samples in this series were weighed accurately on an analytical balance, each to within 0.002 gram of weighing exactly 5 grams. All samples from each piece of material were weighed consecutively, moisture samples being taken after weighing one-third, after weighing two-thirds, and after weighing the last sample. The moisture was determined as the average of the three samples by drying to constant weight in an air oven at 105° ± 1°C. Results, except fluidity values, are computed to the oven-dry weight of sample. Samples for Soxhlet extraction were weighed consecutively in 43-by-123-mm., double-thickness, fat-free extraction thimbles. For the ethyl alcohol-diethyl ether extractions at room temperature, the cotton was weighed in beakers, part of it transferred to wide-mouthed Erlenmeyer flasks, and the beakers reweighed. The difference between the two weighings is the sample weight.

Solvents Used

The solvents used throughout the experiments were as follows:

1. Ethyl Alcohol. The 95-percent azeotropic alcohol-water mixture, distilled from potassium hydroxide (1 gram per liter), the fraction distilling at 78°C, was used without further treatment.

2. Isopropyl Alcohol. Commercial isopropyl alcohol was shaken with 10-percent sodium hydroxide, and allowed to stand until the layers separated. The lower layer was discarded and the fraction distilled from the upper layer at 80°C was used.

3. Methyl Alcohol. The C.P.–A.C.S. absolute methanol, having a boiling range of 64° to 66°C, was used without further purification.

4. Acetone. The C.P.–A.C.S. dimethyl ketone was used without further purification.

5. Benzene. Benzol, C.P.–A.C.S., boiling range 79.5° to 81°C, thiophene-free, was used without further purification.

6. Ether. Specially purified ether suitable for anesthesia containing about 2 percent alcohol and 0.5 percent water was further purified by Werner's method [31]. When thus purified, the ether was found to be free from acids, aldehydes, peroxides, and unsaturated compounds. For the analyses requiring dried ether, the further purified ether was allowed to stand over 4-mesh calcium chloride for 18 to 24 hours before use.

7. Chloroform. C.P. chloroform was purified immediately before use by shaking 5 times with an equal volume of water, as suggested by Cohen [7]. Dried chloroform was obtained by drying over calcium chloride for 18 to 24 hours after distilling at 61° to 61.2°C at 759.6-mm. barometric pressure.

8. Carbon Tetrachloride. A.C.S. reagent-grade carbon tetrachloride, boiling range 76° to 78°C, was used without further purification.

9. Trichloroethylene. Technical-grade material was purified by distillation so that it had a boiling range of 85° to 87°C.

Experimental Data

In the preliminary experiments to determine optimum time, samples were extracted in Soxhlet apparatus with chloroform, ethyl and isopropyl alcohols for periods, measured from the time of the first siphoning, of from 10 minutes to 50 hours. The chloroform extracts were transferred directly to tared 100-ml. beakers. The ethyl and isopropyl alcohol extracts were transferred to separatory funnels and the chloroform-soluble portion separated by the Conrad technique [9], the final extract being transfered to tared beakers. For all three extractions, the extractives were dried in the tared beakers to constant weight in an air oven at 105° ± 1°C.

To determine the amount of degradation of cotton caused by extended Soxhlet extraction with various solvents, duplicate samples were extracted for 100 hours with 95 percent ethyl alcohol, benzene, chloroform, and carbon tetrachloride. The heat under the Soxhlet apparatus was so adjusted that siphonings occurred at 3- to 4-minute intervals. The alcohol-extracted samples were rinsed several times with distilled water to remove the adhering solvent. Alcohol-extracted and rinsed samples, as well as those from the other extractions (without rinsing), were air-dried overnight, cut into small pieces, and then put through a small Wiley mill and a 20-mesh screen. The extent of degradation was measured by comparison of the fluidities determined by the Conrad veloc-

TABLE I. TEMPERATURES OF EXTRACTION

Solvent	Low temperature (°C)	High temperature (°C)
Ethyl alcohol	74.0	76.2
Chloroform	59.0	59.0
Dried chloroform	59.8	60.2
Diethyl ether	34.2	34.6
Dried diethyl ether	34.2	34.6
Alcohol and diethyl ether	24.4	25.5
Benzene	77.5	78.5
Benzene methanol	56.0	56.0
Trichloroethylene	82.2	82.6
Carbon tetrachloride	73.6	74.0
Acetone	55.8	56.0

ity-gradient technique [8]. Fluidity results were computed on the basis of the cellulose contents determined by the Kettering and Conrad wet oxidation with acid-dichromate solutions [16] after the extraction recommended by Reid, Nelson, and Aronovsky [24].

Extraction methods used in the experiments employing uniform conditions (except temperatures, Table I) varied somewhat with the solvent being tested. Conrad techniques for wax extraction [9] were followed except that 100 ml. of distilled water were used in the separatory funnels instead of the 75 ml. specified, since, with this quantity of water, phase separations occur more readily. In the successive ethyl alcohol-diethyl ether extractions, the cottons stood for 24 hours at room temperature in 250 ml. of 95-percent ethyl alcohol. The alcohol was removed on a Büchner funnel using Whatman's No. 50 filter paper and the cotton was replaced in the Erlenmeyer flask. It was then covered with 250 ml. of purified diethyl ether and allowed to stand at room temperature for another 24 hours. The ether was removed as before and the extracts were combined. The benzene-methanol solvent was a mixture of equal volumes of the two reagents. In all Soxhlet extractions, 250 ml. of solvent were added to the Soxhlet flask, and heat was so adjusted that siphonings occurred at 3- to 4-minute intervals. All extracts were transferred to 100-ml. tared beakers, where the solvent was evaporated and the residue dried to constant weight at 105° ± 1°C. Table I gives the high and low temperatures in the extraction chamber read with the assistance of a hand lens from E. and A. Anschütz Standard Centigrade thermometers having an enclosed glass scale in accordance with National Bureau of Standards specifications.

Results

Rate of Extraction

In the preliminary experiments (Figures 1, 2, and 3) a greater quantity of wax was obtained by using the Conrad technique for all extractions up to 30 hours. This technique using ethyl alcohol extracted 0.72 percent of wax in 2 hours, whereas extraction directly with chloroform did not reach that quantity until after 30 hours. Both the chloroform and the ethyl alcohol extracts, after 50 hours' extraction, contained tarry, sticky deposits indicating deterioration of the wax fraction probably due to prolonged heating. The most surprising result was the quantity of extractives obtained after only 10 minutes' extraction. With chloroform this amounted to 70 percent

TABLE II. DAMAGE TO COTTON FABRICS CAUSED BY EXTRACTION WITH VARIOUS SOLVENTS

Identification of print cloth	Extracting solvent	Extraction time (hrs.)	Cellulose content (%)	Fluidity (rhes) C = 0.5 g./100 ml. G = 500 sec^{-1}
No. 1	Alcohol	6*	95.38	2.30
No. 1	Alcohol	100	95.27	2.44
No. 1	Carbon tetrachloride	100	93.48	2.54
No. 2	Alcohol	6*	96.87	6.96
No. 2	Alcohol	100	96.48	7.22
No. 2	Carbon tetrachloride	100	94.96	7.94
No. 3	Alcohol	6*	95.63	2.32
No. 3	Benzene	100	96.48	2.41
No. 3	Dried chloroform	100	94.42	2.44
No. 4	Alcohol	6*	98.80	7.36
No. 4	Benzene	100	97.90	9.50
No. 4	Dried chloroform	100	98.04	8.07

* The 6-hour extraction of control samples is desirable as a preliminary step in the fluidity determinations.

TABLE III. WAXY EXTRACTIVES FROM SELECTED COTTONS OBTAINED WITH DIFFERENT SOLVENTS, EXPRESSED AS PERCENTS OF THE DRY WEIGHT OF SAMPLE

Solvent or technique	Time of extraction (hrs.)	Raw cotton (%)	Broadcloth No. 1 (gray goods) (%)	Broadcloth No. 2 (desized and washed) (%)	Broadcloth No. 3 (kier-boiled, not scoured) (%)	Bagging material (kier-boiled, soap after kier) (%)	Broadcloth No. 4 (hypochlorite bleach) (%)	Broadcloth No. 5 (peroxide kier bleach) (%)
Conrad technique (wax) *	6	1.12	1.40	1.48	1.00	1.04	0.40	0.56
Carbon tetrachloride	6	0.59	1.36	1.26	0.63	0.46	0.34	0.20
Alcohol and ether, successively at room temperature	24 for each	1.08†	1.88†	1.52†	0.82†	0.86†	0.48†	0.36†
Chloroform	6	0.79	1.44	1.34	0.78	0.59	0.40	0.26
Chloroform—dried	6	0.88	1.48	1.54	0.86	0.76	0.54	0.48
Diethyl ether	6	0.86	1.62	1.44	1.00	0.80	0.46	0.49
Diethyl ether—dried	6	0.71	1.54	1.53	0.72	0.32	0.34	0.42
Benzene-methanol mixture	6	1.46†	2.28†	1.88†	1.42†	1.38†	0.70†	0.82†
Benzene	6	0.74	1.40	1.22	0.84	0.66	0.32	0.34
Trichloroethylene	6	0.74	1.54	1.37	0.85	0.73	0.34	0.34
Acetone	6	0.84	0.51	0.32	0.87	0.50	—	0.45

* Alcohol-soluble material is much more than these percentages.
† Contains water-soluble constituents.

of the maximum amount obtained with 30- to 50-hour extraction, and with ethyl alcohol it amounted to 83 percent. This is a larger percentage of the total wax content than is removed by the normal kier-boil and bleach.

Cellulose Degradation

The degradation, in no case excessive, caused by prolonged extraction shows that ethyl alcohol, chloroform, carbon tetrachloride, and benzene, in the order named, are increasingly harmful to fabrics and that the degradation was greater in the samples having the greater initial damage (Table II). The British Fabrics Research Committee [11] classed fabrics having a fluidity of 1 to 5 rhes as "mildly scoured and bleached" and those having a fluidity of 5 to 10 rhes as "normally scoured and bleached." While the values here reported, because of differences in the analytical methods, are not strictly comparable with those of the British Fabrics Research Committee, they are close enough to indicate that none of the samples was excessively damaged in the 100-hour extraction.

Extraction of Uniform Samples by Different Solvents

The comparative percentages of extractives, based on dry weight of sample, obtained from identical cottons by different solvents are shown in Table III. Each of the solvents placed the seven cottons in approximately the same order with regard to quantity of extractives. Solvents which gave nearly complete extraction with some of the cottons, however, apparently failed to approach it with others. Acetone extractives were so unpredictable that caution should be used in selecting this solvent when comparable data are desired. Dried chloroform extracted more than chloroform that had not been dried. Drying diethyl ether reduced the quantity of extractives. Carbon tetrachloride yielded small percentages of extractives with every type of fabric tested. Most noticeable in the results obtained are the variations in the quantities extracted. The benzene-methanol mixture gave the largest quantity of extractives in every case. As should be expected, however, both the benzene-methanol Soxhlet extractions and successive alcohol and ether extractions at room temperature included water-soluble materials. This was demonstrated by taking extracts from raw cotton and from the cotton bagging material which had been

TABLE IV. COMPARISON OF QUANTITIES OF WAX EXTRACTED FROM COTTON BY THREE DIFFERENT SOLVENTS OR SOLVENT COMBINATIONS

Sample	Solvents or Technique: Conrad technique (Soxhlet) %	Benzene-methanol (Soxhlet) %	Alcohol ether (room temperature) %
Raw cotton	1.12	0.91	0.46
Bagging material soaped after kier	1.04*	0.93*	0.55*

* Values may include fatty matter from the soap.

kier-boiled and subsequently soap-scoured and concentrating them to approximately 30 ml. and then separating the waxes from the water-soluble materials by use of the Conrad water-alcohol-chloroform phase separations [9]. It was found that the quantities of waxes obtained were lower than those yielded by the regular Conrad techniques using ethyl alcohol. A comparison of the values is given in Table IV.

The Conrad technique for determining wax has been found to be more efficient than any other technique tried when water-soluble substances are excluded. It is doubtful that any one solvent will remove all the wax from cotton within a reasonable length of time. Hot ethyl, or isopropyl, alcohol gives more nearly complete extraction, however, than any other solvent tried. The effectiveness of ethyl alcohol in removing wax from cotton may be due to its ability to mix readily with both hydrophobic and hydrophilic materials. In general, the differences in quantity of wax extracted by various solvents may possibly be due to poor penetration, selective solubilities of the solvent used, or to inaccessibility of the wax.

The extractives from the trichloroethylene solvent were black and tarry in appearance and had the typical fragrant odor of organic esters. Seventy-five milliliters of trichloroethylene added to a number of dried extractives from other solvents with the extracts again dried to constant weight also resulted in the tarry appearance and sweetish, fragrant odor. Furthermore, in almost every case, there was an increase in weight. This solvent, therefore, was not considered to be satisfactory for wax determinations.

A comparison of the quantities of extractives obtained with solvents, other than benzene-methanol, indicated the solvents to be decreasingly effective in the order named: the Conrad technique using alcohol and chloroform; dried chloroform; diethyl ether; alcohol followed by ether, cold; acetone; dried diethyl ether; chloroform; and benzene. The order, however, differed with each type of fabric treatment. It would appear advisable, therefore, to consider the type of treatment the fabric had received before selecting the solvent for extraction.

When the Conrad technique was used with raw cotton, the wax percentage was larger than the total extractives obtained with any of the other solvents except the benzene-methanol mixture, which, of course, contained water-soluble constituents. When the extracts from successive 24-hour alcohol and ether treatments at room temperature were combined

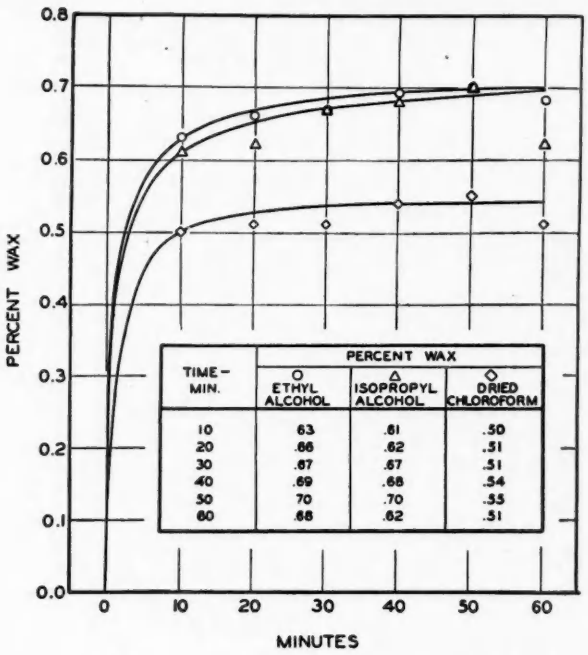

FIG. 1. *Wax from Empire cotton by the Conrad techniques using ethyl and isopropyl alcohols compared with those obtained by Soxhlet extraction using chloroform.*

the yield was nearly, but not quite, as much as the wax obtained with the Conrad technique. Hence, since these extractives also contain water-solubles, the cotton successively extracted at room temperature [27] was probably not completely "dewaxed."

When the raw cotton was ground through a Wiley mill and thoroughly mixed, the quantities of wax extractives obtained by the Conrad technique, chloroform, and dried chloroform extractions were 1.12, 0.79, and 0.88 percent, respectively (Table III). The highest quantities from the same cotton obtained after the same time of extraction when the cotton had not been previously ground were 0.72 percent with the Conrad technique and 0.61 percent with dried chloroform (Figure 2). The increased amounts of extractives represent real differences which cannot be traced entirely to variations in techniques or experimental conditions but may possibly be partly caused by increased solvent penetration made possible by the grinding of the cotton. Knecht and Streat [19] report that grinding cotton increased the quantity of wax extract. These increases may not necessarily be general since data obtained in this laboratory on another cotton indicate considerably smaller increases due to grinding. Further investigations of this phenomenon are now in progress.

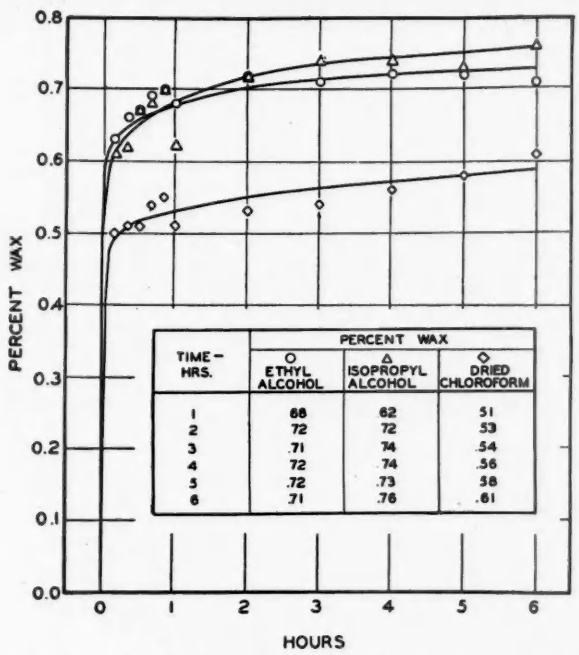

FIG. 2. *Wax from Empire cotton by the Conrad techniques using ethyl and isopropyl alcohols compared with those obtained by Soxhlet extraction using chloroform.*

With gray goods, the carbon tetrachloride and acetone were the only solvents yielding quantities of extractives lower, and diethyl ether the only one yielding more, than the Conrad technique. Other differences in the values obtained are apparently not significant.

The high wax values for the desized fabric are in line with those usually found in this laboratory. Desizing agents probably fail to remove all the extractable constituents added with the size and may even add to that already present on the fabric.

Except for the differences already discussed, no striking irregularities appear in solvent extracts from fabrics kier-boiled, kier-boiled and soaped after the kier, or bleached with sodium hypochlorite or hydrogen peroxide.

Conclusions

Different solvents extracted widely different amounts of waxy materials in experiments where quantity of solvent and sample, rate and total time of extraction, and other laboratory conditions were held constant. The Conrad technique using either ethyl or isopropyl alcohol was more efficient than any other technique tried for extraction of waxy materials from cotton. Trichloroethylene is an unsuitable solvent for use in cotton-wax determinations. A 6-hour extraction period was found to be adequate for comparative purposes. Ethyl alcohol, chloroform, carbon tetrachloride, and benzene, in the order named, were increasingly harmful to cotton cloth in a 100-hour extraction experiment. However, since these solvents produced only slight degradation in the extracted fabrics, even in that length of time, all of them may be safely used in Soxhlet extraction of cotton fiber, yarn, or fabric.

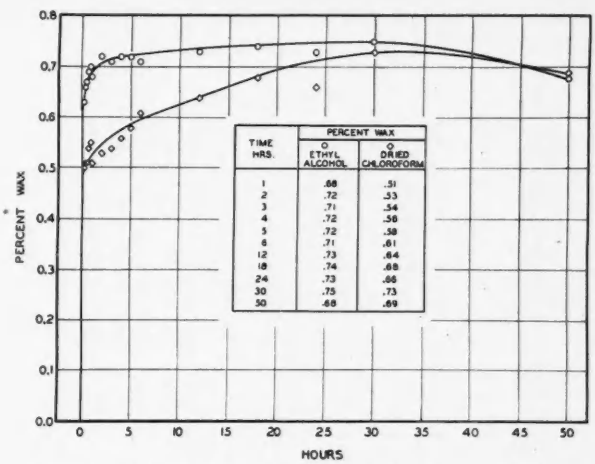

FIG. 3. *Wax from Empire cotton by the Conrad techniques using ethyl alcohol compared with those obtained by Soxhlet extraction using chloroform.*

Acknowledgment

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Mechanical Properties of Textiles, XI*

Application of the Theory of the Three-Element Model to Stress-Strain Experiments on Cellulose Acetate Filaments

C. H. Reichardt† and Henry Eyring‡

Contribution of The Textile Foundation and Princeton University, Frick Chemical Laboratory, Princeton, New Jersey

I. Introduction

Two experimental methods of examining the mechanical properties of fibers—viz., stress-strain measurements at constant rate of elongation and creep measurements at constant load—have been analyzed in previous papers of this series on the basis of the three-element model. Paper IV [3] first analyzed a number of experiments on viscose rayon fibers on the basis of the three-element model. Paper X [5] discussed the creep curves of cellulose acetate on the basis of two equivalent three-element models, which here are reproduced in Figure 1. As was stated in Paper X, creep curves are explained best on the basis of Figure 1B, while stress-strain curves at constant rate of elongation are explained best on the basis of Figure 1A. It is the object of this paper to show the correlation of the two equivalent models.

Following the methods described in Papers I, III, and IV [1, 2, 3], in examining a typical stress-strain experiment at constant rate of elongation, as given in Figure 2, on the basis of Figure 1A, the full curve represents the total force on the complete model, whereas the dashed curve gives the force on the open spring, s_2, throughout the experiment. The difference between the two curves represents the force on the Maxwell element as represented by its working curve (Figure 4). As the fiber is first elongated in a stress-strain experiment, the total force on the fiber increases rapidly. The dashpot has not yet started to flow. The two springs, s_1 and s_2, are elongating and the initial slope is thus the sum of the two spring constants, k_1 and k_2. Thus,

$$\left(\frac{dF}{dl}\right)_{\text{initial}} = k_1 + k_2. \tag{1}$$

As the fiber is further elongated it begins to yield—that is, the dashpot is now flowing at the same rate that the fiber is being elongated, the spring, s_1, is no longer elongating, and the final slope is equal to the spring constant, k_2, of the open spring, s_2, and

$$\left(\frac{dF}{dl}\right)_{\text{final}} = k_2. \tag{2}$$

Reversing the apparatus, so that the fiber is now contracting, the springs are relaxing, the dashpot is contracting, and the curve is steeper than the initial slope on elongation. After a time the dashpot is no longer moving, the two springs alone are relaxing, and the slope is equal to the initial slope on elongation.

If the curve in Figure 2 is now examined in the

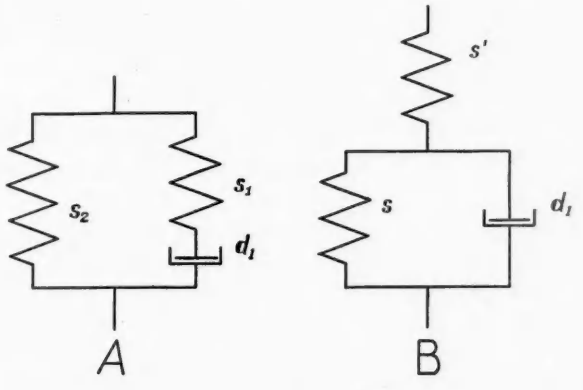

FIG. 1. *Three-element model. (This is identical with Figure 2, Paper X, in which the symbols s_1 and s_2 should be transposed.)*

* This is the eleventh paper in the series "The Mechanical Properties of Textiles." The previous papers were published in the September and December, 1945, and the January, February, March, May, July, and August, 1946, issues.

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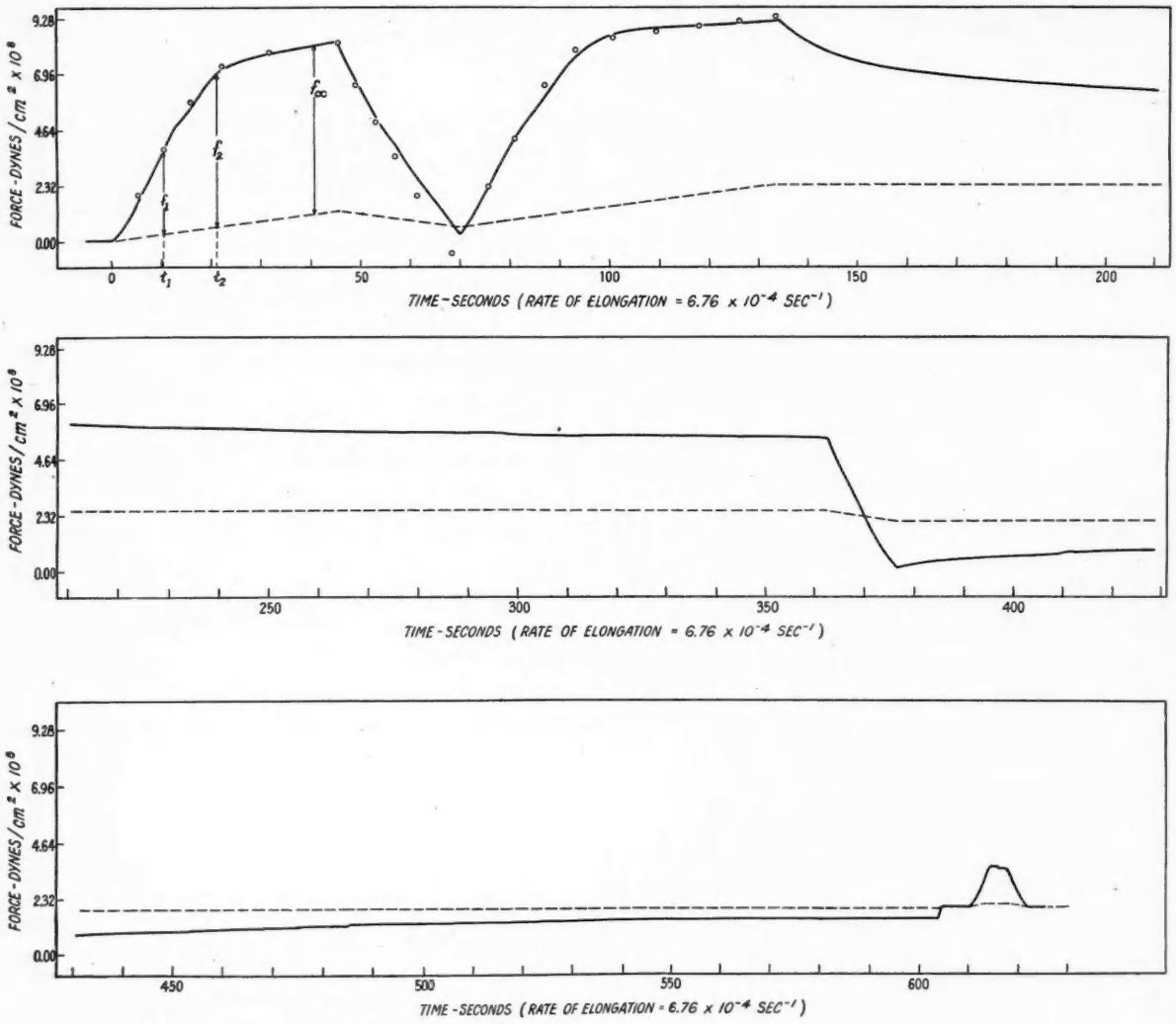

FIG. 2. *Typical stress-strain curve as obtained on the Sookne-Rutherford autographic balance (Fiber 1).*

light of the model in Figure 1B, it is seen that when the fiber is first elongated, since the dashpot is not yet flowing, the initial increase in force is due to the elongation of the spring, s'. The initial slope is thus equal to the spring constant, k', of the spring, s', and relating this to the preceding paragraph it is seen that

$$\left(\frac{dF}{dl}\right)_{\text{initial}} = k_1 + k_2 = k'. \tag{3}$$

As the fiber yields, the dashpot, as before, is starting to flow at the same rate that the fiber is being elongated and the final slope is attributable to the elongation of both springs s and s'. Since these springs are in series, the reciprocals of the spring constants are additive and they can be related to those of the other model by

$$\frac{1}{\left(\frac{dF}{dl}\right)_{\text{final}}} = \frac{1}{k_2} = \frac{1}{k} + \frac{1}{k'}. \tag{4}$$

II. Method of Analysis

The solid line in Figure 2 is a typical stress-strain curve for cellulose acetate as obtained on the Sookne-Rutherford autographic balance. A complete stress-strain experiment on any fiber actually consists of a series of such curves obtained at different rates of elongation for each curve. To prepare this curve for analysis, it is convenient to transfer

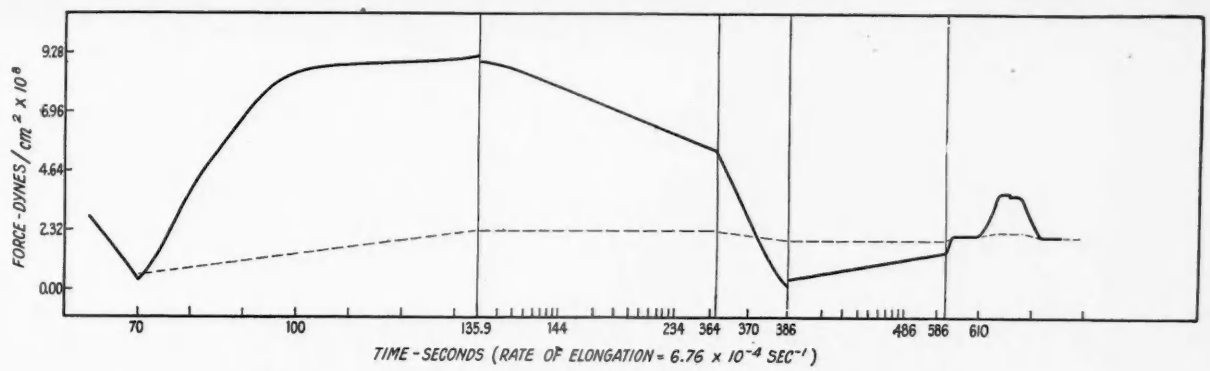

FIG. 3. *Figure 2, with the relaxation curves on a logarithmic scale.*

the long relaxation curve obtained on the apparatus to a logarithmic scale. The latter portion of the stress-strain curve reproduced in Figure 2 is again reproduced in Figure 3, with the relaxation curves on a logarithmic scale. All other curves reproduced in this paper will appear in this latter form.

The force on the open spring, s_2, must then be established throughout the experiment. In the previous section, it was pointed out that the final slope of the curve is attributable to the open spring; hence this force can be determined at any point in the experiment by tracing the point of ultimate relaxation of the fiber throughout the experiment with this final slope. In this paper the dashed curves represent this force on the open spring s_2. During an elongation the force on the open spring is increasing; during a contraction, it is decreasing with this same slope; while during a relaxation, it is remaining constant. This dashed curve should, as pointed out in previous papers, cut the experimental curve through the points of constant slope; that is, the angles it makes with the experimental curve should be equal throughout the experiment for a given rate of elongation. This open spring force curve should also pass through the origin of the experimental curve; that is, in a series of experimental curves this dashed curve should connect all of the points of ultimate relaxation. The distance between the experimental curve after yielding and the dashed curve, which represents the force, f_∞, on the Maxwellian element, should also be equal for every elongation throughout an experiment at any one rate of elongation. If these conditions do not hold, then the three-element model is not applicable and a method similar to that described in Paper IX [4] must be used to explain the behavior of the fiber.

1. The Spring Constants

In the experiment given in Figures 2 and 3, the rate of elongation of the apparatus was 4.8×10^{-3} cm/sec, and as the length of the fiber under analysis was 7.1 cm, the rate of elongation of the experiment, ρ, is

$$\rho = \frac{4.8 \times 10^{-3}}{7.1} = 6.76 \times 10^{-4}\ \text{sec}^{-1}. \qquad (5)$$

The radius of the fiber, as calculated from the denier and the density, was 9.5×10^{-4} cm. As the distance between the two curves after yielding (f_∞ in Figure 2) is equivalent to a load of 2.016 grams, the load, a, on the Maxwellian element is given by

$$a = \frac{Gf_\infty}{\pi r^2} = \frac{980 \times 2.016}{\pi(9.5 \times 10^{-4})^2} = 6.96 \times 10^8\ \text{dynes/cm}^2 \qquad (6)$$

and

$$\alpha = \frac{\log 2\beta}{a} = \frac{8.3}{6.96 \times 10^8} = 1.19 \times 10^{-8}\ \text{cm}^2/\text{dyne}.$$

The spring constants are determined from the initial and final slopes through equations (3) and (4). Thus for Figure 2

$$\left(\frac{dF}{dl}\right)_{\text{initial}} = k' = k_1 + k_2 = 4.77 \times 10^{10}\ \text{dynes/cm}^2 \qquad (3)$$

and

$$\left(\frac{dF}{dl}\right)_{\text{final}} = k_2 = 3.72 \times 10^9\ \text{dynes/cm}^2. \qquad (7)$$

Therefore from equation (4)

$$\frac{1}{k} = \frac{1}{k_2} - \frac{1}{k'} = \frac{1}{3.72 \times 10^9} - \frac{1}{4.77 \times 10^{10}}$$

and

$$k = 4.05 \times 10^9\ \text{dynes/cm}^2.$$

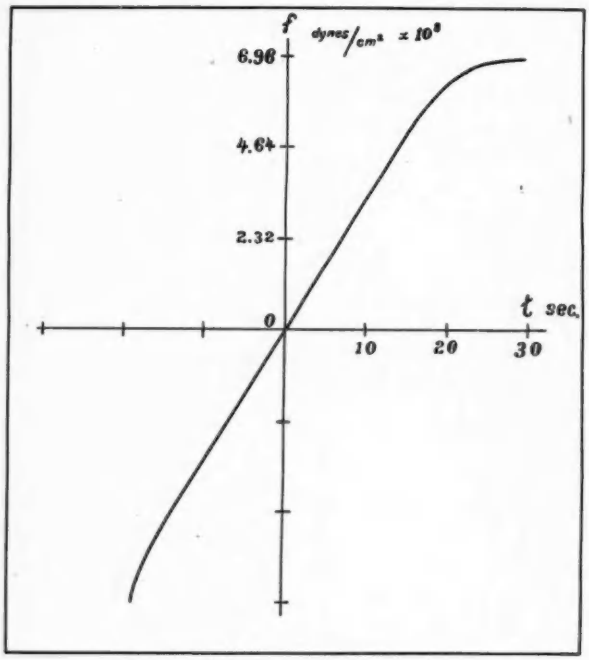

FIG. 4. *Working curve for Figure 2.*

2. The Viscous Constants

The viscous constant, β, can be determined by any one of several methods:

(*a*) The experimental curve at the yield point is dependent on β. When β is large, the yield point becomes sharper or more definite, while for small β the yield point is very gradual. Following the method of Paper I [1], Section IV, *B*, the value of β can be calculated by determining the force, f_2, on the dashpot at the time, t_2, which is twice the time, t_1, it takes the force to reach one half of the final value, f_∞. Thus in Figure 2 the total force on the dashpot is found to be 2.016 grams. One half of that load is reached at $t_1 = 10.4$ seconds. The force, f_2, at twice this time is found to be equal to 1.85 grams, or, in reduced units,

$$f_{2\,\text{red.}} = \frac{f_2}{f_\infty} = \frac{1.85}{2.016} = 0.918. \tag{8}$$

From Figure 5, using this reduced value of f_2, β is determined.

$$\beta = 2{,}000.$$

(*b*) The second method of determining β arises from the relaxation curve, which is a function of the Maxwellian element alone. The value of the reduced force, f_2, acting on the dashpot at a time t_2 is obtained from the logarithmic relaxation curve (t_2 is twice the time, t_1, it takes the force to reach one half of its initial value, f_∞). Following the method described in Paper I [1], Section IV, *C*, it is seen that for the first relaxation curve in Figure 3

$$f_{2\,\text{red.}} = 1 - \frac{f_2}{f_\infty} = 1 - \frac{1.180}{2.016} = 0.414. \tag{9}$$

And from Figure 5, Paper X [5],

$$\alpha = 8.24.$$

Since, for large β

$$\alpha = \log 2\beta, \tag{10}$$
$$\beta = 1{,}900.$$

(*c*) A method of determining β that does not depend on the shape of the curve but is determined from scale factors alone is obtained from a series of experiments at differing rates of elongation. The equation governing the ratio

$$R = \frac{a_n}{a_1}, \tag{11}$$

where a_1 refers to the value of f_∞ at the slower rate and a_n to the rate n times as fast, given in the approximate form for large values of β, is

$$\beta = \tfrac{1}{2} n^{\left(\frac{1}{R-1}\right)}, \tag{12}$$

TABLE I.

1	2	3	4	5	6	7	8	9	10
Experiment	Fiber	ρ ($\text{sec}^{-1} \times 10^{-4}$)	l (cm)	k' ($\frac{\text{dynes}}{\text{cm}^2} \times 10^{10}$)	k ($\frac{\text{dynes}}{\text{cm}^2} \times 10^{9}$)	k_1 ($\frac{\text{dynes}}{\text{cm}^2} \times 10^{10}$)	k_2 ($\frac{\text{dynes}}{\text{cm}^2} \times 10^{9}$)	β (1st slope)	β (2nd slope)
1	1	6.76	7.1	4.77	4.05	4.40	3.72	2,000	1,000
2	2	6.91	6.95	4.37	2.82	4.10	2.65	1,000	250
3	2	1.73	6.95	5.25	2.79	4.99	2.65	600	500
4	12	1.67	7.2	4.52	4.10	4.15	3.77	600	600
5	12	6.67	7.2	4.52	4.10	4.15	3.77	2,000	600
6	12	0.267	7.2	4.60	4.20	4.22	3.84	10	50
7	12	6.67	7.2	4.52	4.10	4.15	3.77	150	200
8	32	6.67	7.2	5.28	4.07	4.91	3.77	1,000	1,000
9	32	0.185	7.2	4.74	3.72	4.39	3.45	13	13
10	3	7.28	6.6	5.53	3.14	5.23	2.97	2,500	1,000
Paper X	—	0.0	—	3.54	4.35	—	—	—	—

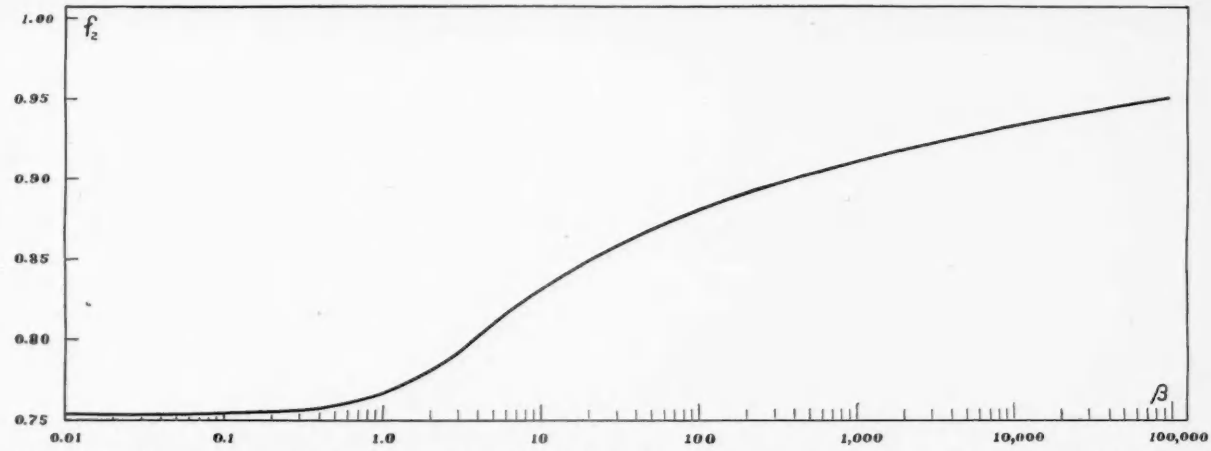

FIG. 5. *Diagram for evaluating β from the reduced force, f_2, acting on the dashpot at a time, t_2, which is twice the time elapsed from the application of the load until it reaches one half its final value. (The upper portion of the original graph, Figure 9, Paper I, was in error. The corrected graph is reproduced above.)*

where β refers to the slower rate. β for the faster rate is n times β for the slower rate.

3. The Theoretical Curve

The working curve, Figure 4, for the Maxwellian element for each value of β is calculated from the equation,

$$\ln\left\{\frac{1+\frac{\beta+1}{\sqrt{\beta^2+1}}\tanh At}{1+\frac{1-\beta}{\sqrt{\beta^2+1}}\tanh At}\right\} = \frac{}{\ln\left\{\frac{1+\frac{\beta+1}{\sqrt{\beta^2+1}}}{1+\frac{1-\beta}{\sqrt{\beta^2+1}}}\right\}},$$

by substituting the proper values of f and t for the reduced terms obtained.

The theoretical curve is then constructed using this working curve and the force on the open spring, as indicated by the dashed curve. The exactitude of the fit of the theoretical curve to the experimental is naturally an excellent criterion for the proper value of the viscous constant β, and thus it is another method of determining the value of β.

The rate constant, K, of the dashpot is evaluated from the value for β and the rate of elongation, ρ, since

$$K = \frac{\rho}{\beta}. \tag{14}$$

The volume of flow, V_f, is calculated from the equation

$$V_f = 2k_0T\alpha, \tag{15}$$

where k_0 is the Boltzmann constant, T is the absolute temperature, and

$$\alpha = \log 2\beta. \tag{10}$$

TABLE I.—*Continued*

11	12	13	14	15	16	17	18	19
β (↓ relaxation)	β (↑ relaxation)	β (rate)	β (typical)	a $\left(\frac{\text{dynes}}{\text{cm}^2}\times10^8\right)$	α $\left(\frac{\text{cm}^2}{\text{dynes}}\times10^{-8}\right)$	K ($\text{sec}^{-1}\times10^{-7}$)	V_f (Å. cubed)	$\Delta F^{\pm}$ (cal.)
1,900	1,800	—	2,000	6.96	1.19	3.38	9.91	26,300
2,000	2,000	2,000	2,000	6.40	1.30	3.45	10.22	26,300
500	500	550	500	6.05	1.14	3.46	9.78	26,300
500	500	400	500	5.23	1.32	3.34	10.28	26,300
1,500	2,500	1,600	2,000	5.81	1.43	3.34	10.55	26,300
16	—	27	80	3.49	1.45	3.34	10.62	26,300
2,000	—	1,600	2,000	5.81	1.43	3.34	10.55	26,300
2,000	2,000	3,240	2,000	6.96	1.19	3.33	9.91	26,300
150	—	90	55	4.43	1.06	3.38	9.55	26,300
1,800	140	—	2,000	8.48	0.97	3.64	9.26	26,300
—	—	—	—	6.20	1.52	0.0879	10.70	29,800

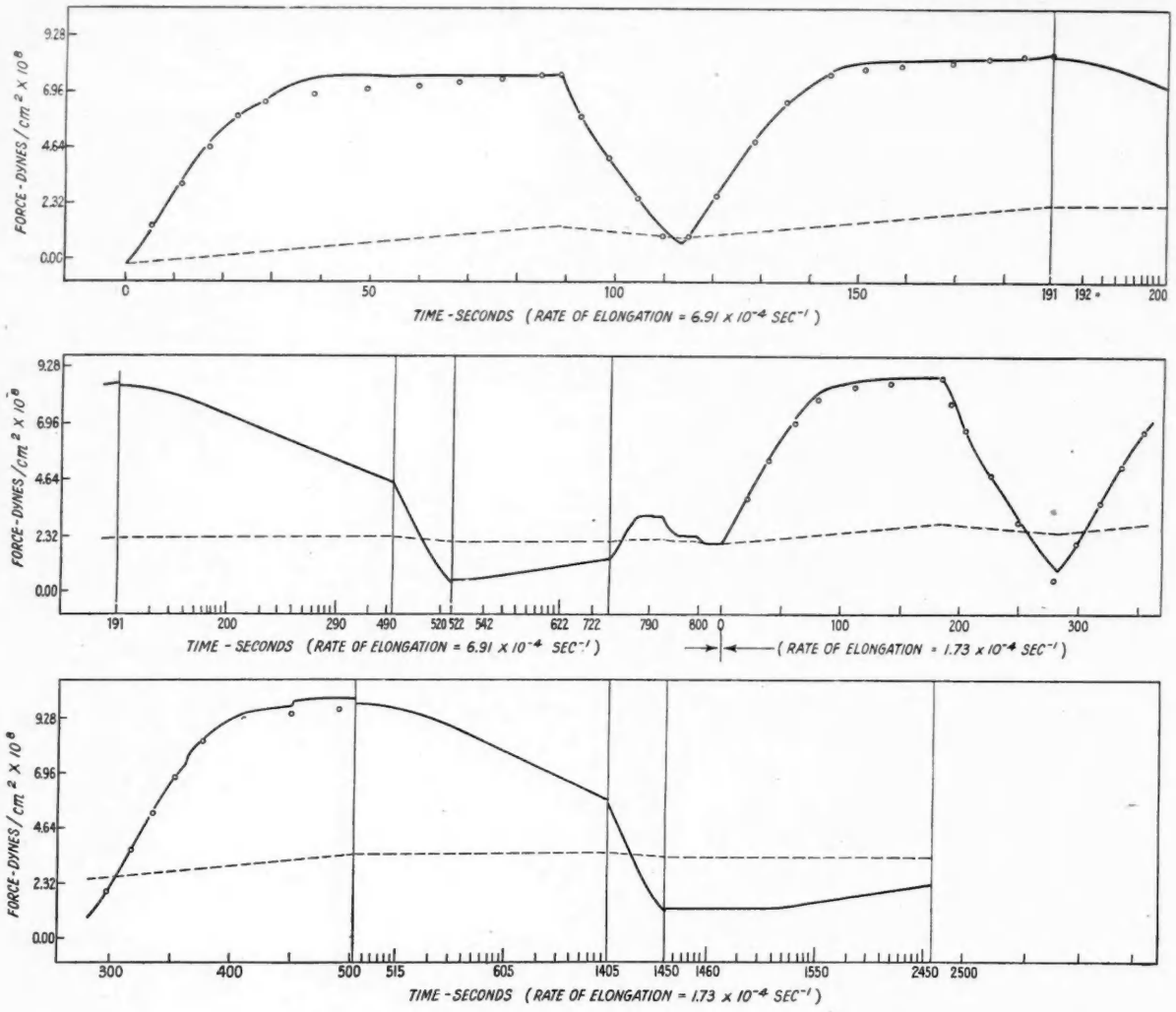

FIG. 6. *Stress-strain curve of Fiber 2.*

III. Discussion of Results

The data in Table I were taken as typical from a series of experiments on 3.5-denier cellulose acetate filaments conditioned at 56.9 percent relative humidity. The experiments were conducted at room temperature, 27°C. The spring constants of different filaments and of the same filament at differing rates of elongation are in good agreement with one another. It is seen that these results are also in good agreement with the results of Steinberger on the creep of 3.75-denier cellulose acetate filaments at 60 percent relative humidity, which were discussed in Paper X and are reproduced in Table I.

The values of viscous constant β are not in as good agreement with one another as are the spring constants. Unlike the spring constants, β is proportional to the rate of elongation. The values of β given in the first two columns (9, 10) of Table I were calculated by the first method described previously (*i.e.*, from the shape of the stress-strain curve). These values can be expected to vary greatly, as small fluctuations in the experimental curve at the point, t_2, would cause large errors in β. The next two columns (11, 12) of β values were calculated by the second method (*i.e.*, from the relaxation curve). This method should, and does, give better β values than that dependent on the shape of the curve as it is measured over longer periods of time and small mechanical fluctuations are canceled out. In a few of the filaments tested, particularly after they had been exposed to several cycles of tests, the upward relaxation gave low values of β (column 12, Table I),

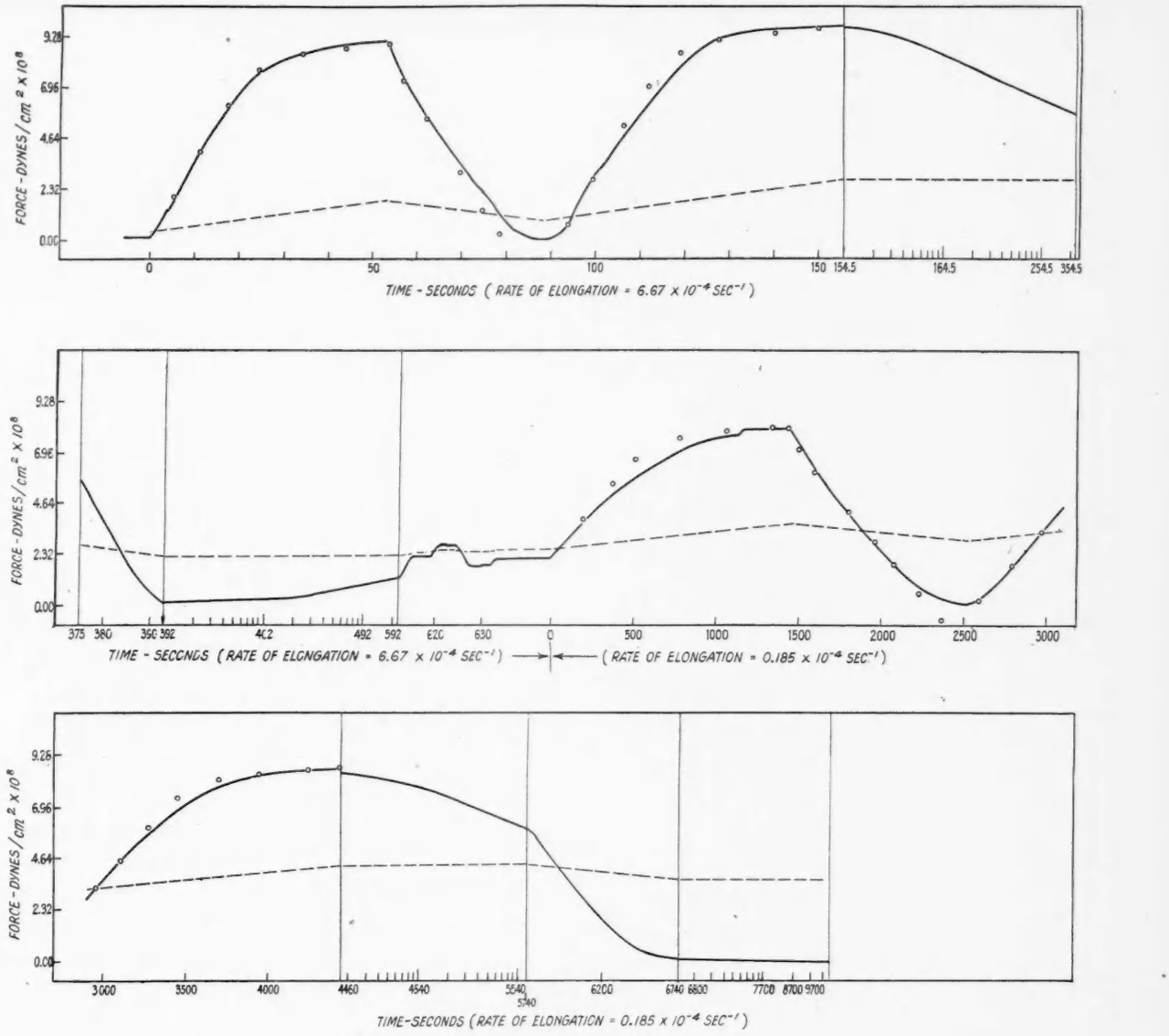

FIG. 7. *Stress-strain curve of Fiber 32.*

indicating poor recovery power in cellulose acetate. In one case at a very slow rate of elongation, the force on the fiber did not increase at all as it generally does. Column 13 gives the β values calculated by the third method (*i.e.*, ratio of the total force on the dashpot at differing rates of elongation); this is also a more accurate method of determining β than that depending on the shape of the stress-strain curve. Column 14 lists the values of β which were taken as being average for that rate of elongation; those values of β were used in fitting the experimental curves and in calculating the other viscous constants. The points on Figures 2, 3, 6, and 7 are theoretical and are calculated on the basis of these values of β and the spring constants. That the experimental curves are in good agreement with the theoretical can be seen.

In discussing Steinberger's creep data (Paper X [5]) it was noted that at higher relative humidities the load used was too heavy; at lower relative humidities the loads were too small. The experiment at 60 percent relative humidity was carried out perhaps with the ideal load. In comparing that experiment with the work in this paper, it is noticed that the load, a, on the Maxwellian element as obtained here is approximately equal to the load Steinberger used in his run. Thus it is conceivable that the criterion for the load to use in creep experi-

ments is the load on the Maxwellian element obtained from stress-strain experiments.

The tendency of cellulose acetate to give low values of β from the upward relaxation curves, particularly after several cycles of tests on one fiber, and the tendency not to have any upward relaxation at all in experiments at low rates of elongation are experimental manifestations of the known poor recovery power of cellulose acetate. This tendency indicates another reason why the relaxation curves give more reliable information of the viscous constants than does the shape of the stress-strain curve.

Acknowledgment

The authors wish to thank Dr. F. Bonnet of the American Viscose Corporation for supplying the acetate samples and to thank Mr. George Halsey for his advice in preparing this paper.

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ABSTRACTS

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A list of periodicals, the abbreviations used in references to articles, and the addresses of publishers of English language publications is given on page 84 of the February, 1946, issue.

ANALYSIS: TESTING: LABORATORY METHODS

★

Testing Chemical Resistance

Apparatus for studying the destruction of textiles by chemicals. I. G. Nagatkin and V. S. Artemova. *Tekstil. Prom.* **5**, No. 11/12, 56 (1945) (through *Chem. Abstr.* **40**, 5571[1] (Sept. 20, 1946)).

A tube drawn to a capillary at the upper end is fitted with a perforated rubber stopper at the lower end. A 3-way stopcock admits chemical to the tube (or draws it off) through the rubber stopper. The sample to be tested is fastened with one end to the rubber stopper and with the other to a thread, which passes through the capillary, over a pulley, and supports a weight. After the chemical is admitted the time is noted when the tested specimen breaks under the load.

Text. Research J. Dec. 1946

Standard Color Cards

Spectrophotometric and colorimetric determination of the colors of the TCCA standard color cards. G. Reimann, D. B. Judd, and H. J. Keegan. *Am. Dyestuff Reptr.* **35**, P323–9, P333–4 (July 1, 1946); cf. *T.R.J.* **16**, 344 (July 1946).

The colors of the two standard color cards, the United States Army Color Card and the Standard Color Card of America, have been determined by basic spectrophotometric and colorimetric procedures. From this study there have been found daylight reflectance, Y, I.C.I. chromaticity coordinates (x, y), Munsell book notations and renotations, and ISCC-NBS color designations. These results permit the TCCA standards to be correlated with the American War Standard Z44–1942.

Text. Research J. Dec. 1946 Authors

Determination of Polymer Density

Use of density-gradient tube in the study of high polymers. R. F. Boyer, R. S. Spencer, and R. M. Wiley. *J. Polymer Sci.* **1**, 249–58 (Aug. 1946).

A density-gradient tube is a vertical glass cylinder in which a vertical density gradient of any desired magnitude can be maintained in a stable condition for prolonged periods by correct choice of pairs of miscible liquids of different densities. The authors have found that the density of polymer particles can be quickly determined by this apparatus, using the floating method. These density measurements have been used to study rates of crystallization of polymers, the homogeneity of copolymers, and rates of polymerization. It is suggested as a means of determining molecular-weight distributions. E. D. Klug

Text. Research J. Dec. 1946

Wettability of Fibers

New dynamic method for determining the wettability of fibers. R. Fal'kovich. *Tekstil'naya Prom.* **6**, No. 3, 25 (1946) (through *Chem. Abstr.* **40**, 5925[5] (Oct. 10, 1946)).

A 5-gram bundle of fibers is suspended from one end of a beam the other end of which moves along an upright scale. The fibers are weighted with 20 grams and are submerged in distilled H_2O. Readings are taken periodically and are calculated in terms of completely wetted fibers taken as 100%.

Text. Research J. Dec. 1946

CHEMICAL AND PHYSICAL RESEARCH

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Theory of Acetate Spinning

A theory of the process of spinning in the production of artificial fibers, especially the spinning (with stretching) of cellulose acetate. Arnulf Sippel. *Z. Elektrochem.* **50**, 152–63 (1944) (through *Chem. Abstr.* **40**, 5245[5] (Sept. 10, 1946)).

Simple formulas are established for the rate of diffusion of acetone from the fiber in the precipitating bath and the change of fiber radius during stretching. From these relationships, formulas are derived which

relate the speed of spinning, bath length, thread number, spinneret radius, stretching, and concentration of the spinning solution. The use of the formulas in predicting the effect of changes in the operations is indicated.

Text. Research J. Dec. 1946

Diffusion Constant of Cellulose

Sedimentation and diffusion of cellulose molecules. J. J. Hermans. *J. Polymer Sci.* **1**, 233–6 (Aug. 1946).

Sedimentation and diffusion constants at infinite dilution for various samples of cotton, ramie, and wood pulp are given. It is found that the sedimentation constant is proportional to $N^{\frac{1}{2}}$ and the diffusion constant is proportional to $(1 + N^{\frac{1}{2}})/N$, where N represents the number of statistical chain elements in the chain in Kuhn's sense. It is concluded that the cellulose chains are randomly coiled structures in solution. E. D. Klug

Text. Research J. Dec. 1946

Nitrogen Derivatives of Cellulose

β-aminoethoxyl- and β-cyanoethoxylcellulose acetates. Thomas S. Gardner. *J. Polymer Sci.* **1**, 289–92 (Aug. 1946).

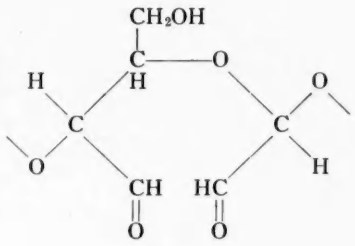
β-aminoethoxylcellulose acetate was prepared by reacting cellulose diacetate with ethyleneimine for 4 hrs. at 100°C under pressure. The product contained 0.19 aminoethoxyl group/glucose unit. A similar product was prepared with cellulose acetate propionate. β-cyanoethoxylcellulose, prepared by refluxing acrylonitrile with alkali cellulose, had a degree of substitution of about 2.3 but was incompletely soluble in organic solvent. Various samples of β-cyanoethoxylcellulose acetate were prepared by reacting cellulose acetate with acrylonitrile in the presence of (1) aqueous NaOH, (2) pyridine, (3) trimethylamine and by acetylating β-cyanoethoxylcellulose in pyridine. Many of these products were soluble in organic solvents. Films of all of these ether esters were dyed with a typical wool-type dye, xylene brilliant blue. E. D. Klug

Text. Research J. Dec. 1946

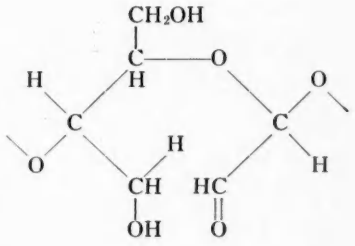
Oxidation of Cellulose

The oxidation of cellulose with buffered periodic acid and the isolation of decomposition products of the oxidized cellulose. Georg Jayme and Spyros Maris. *Ber.* **77B**, 383–92 (1944); cf. *C.A.* **38**, 2197[7] (through *Chem. Abstr.* **40**, 5242[5] (Sept. 10, 1946)).

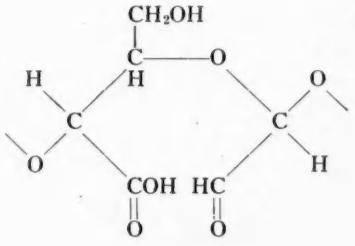
The oxidation of cellulose with buffered HIO_4 (*I*) soln. is studied to obtain new decompn. products in high yield. The course of the oxidation of cotton linters (*II*) with buffered and unbuffered *I* proceeds in a way similar to that of xylan (cf. *C.A.* **38**, 1213[6]). With the buffered *I*, the oxidation occurs more rapidly until the theoretical amt. of *I* is consumed, then the reaction slows down considerably; with the unbuffered soln., the oxidation proceeds more slowly and continues even after the theoretical amt. of *I* has reacted. With the buffered *I*, considerably higher yields of oxidation products are obtained; the fibers shrink markedly even the 1st day; this indicates that the reaction takes place uniformly throughout the fiber, whereas with the unbuffered *I*, the reaction progresses from layer to layer. When the oxidation is carried out in 2 stages, with 10% buffered *I* for 6 days at room temp., then with a 6% *I* for 3 days, 97.5% (theoretical, 98.76%) polymeric aldehyde (*III*) is obtained (cf. Jackson and Hudson, *C.A.* **32**, 5246[1]). *III* is subjected to the following reactions: (*a*) hydrolysis, (*b*) oxidation with Br followed by hydrolysis, and (*c*) hydrogenation in alk. soln. in the presence of Raney Ni to the polymeric aldehyde alc. (*IV*). Kier-boiled and bleached *II* (99.48% α-cellulose, 0.24% ash, Cu no. 0.7, MeOH—C_6H_6 ext. 0.22%, polymerization degree 775, dry content 94.29%) (6 g.) is kept in 93 cc. 9.6% *I* soln. contg. 7.91 g. $AcONa \cdot 3H_2O$ for 6 days at room temp. After this the cellulose is filtered, washed with 4 l. distd. H_2O, and air-dried. Yield 99%. Reduction power according to Bertrand is 54.5% Cu. This product (4.9 g. absolutely dry) is treated in the same way with 83.5 cc. of a 5.3% *I* contg. 3.95 g. $AcONa \cdot 3H_2O$ for 3 days. The



(*III*)



(*IV*)



(*VII*)

filtered, washed, and air-dried *III* has a reduction power of 62% Cu. *III* (3 g.) is refluxed for 12 hrs. with 120 cc. H_2O, filtered, enough *N* HCl or *N* H_2SO_4 is added to give 150 cc. of an 0.1 *N* acid soln., and the mixt. heated for 17 hrs. After removal of the acid with Ag_2CO_3 or $BaCO_3$ resp., 53.5% glyoxalphenylosazone (*V*), m. 167°, is found; glyoxalbenzylphenylosazone, light-yellow needles, m. 197°. The D-erythrose is not detd. in this expt. To a suspension of 11.2 g. *III* in 91 cc. distd. H_2O, 13.7 cc. Br is dropped with stirring, the mixt. kept for 4 days at room temp., and the excess Br removed *in vacuo* at 30°. Half of the mixt. is neu-

tralized with $Ba(OH)_2$ to give 10.64 g. of a Ba salt (*VI*), contg. 31.6% Ba. On decompn. of *VI* with H_2SO_4, 66% polymeric aldehyde acid (*VII*) is obtained as a light-brown resin, very sol. in H_2O. In a 2nd expt. in which the oxidation with Br is carried out for 5 days, 95.1% *VII* (based upon dry *III*) is obtained. Hydrolysis of *VII* by refluxing with 0.1*N* HCl for 20 hrs., and treatment of an aliquot part of the soln. with $PhNHNH_2$ (*VIII*) give 39.8–48% *V*. After removal of the excess *VIII* in the filtrate with BzH, the filtrate is neutralized with $Ba(OH)_2$, and evapd. to dryness to give 4.307 g. Ba salt, contg. 47.4% Ba. This is decompd. with the calcd. amt. of H_2SO_4 and converted into 68% brucine salt of D-erythronic acid, m. 210–12°, $[\alpha]_D^{20} - 22.5°$ (H_2O). Reduction of *III* (9.4 g. absolutely dry) in 219 cc. H_2O and 0.18 g. $Ba(OH)_2$ in the presence of Raney Ni (*IX*) from 0.5 g. Ni—Al, for 174.5 hrs. with intermittent addn. of fresh *IX* and $Ba(OH)_2$, causes the absorption of 2498 cc. H. After removal of the Ba with H_2SO_4, the filtered soln. is hydrolyzed with HCl, and up to 39% inactive erythrol is isolated as the dibenzylidene deriv., m. 197°.

Text. Research J. Dec. 1946

Oxidation of Cellulose

Chemical conversion of macromolecules of cellulose under the influence of oxidizing agents. I. The stability of the glucose bonds in cellulose. E. D. Stakheeva-Kaverzneva and V. I. Ivanov. *Bull. acad. sci. U.R.S.S., Classe sci. chim.* **1945**, 603–8 (in English, 608); cf. *C.A.* **39**, 1755[9] (through *Chem. Abstr.* **40**, 5244[4] (Sept. 10, 1946)).

Monotrityl cellulose ethers, in which only the primary OH is substituted, are not oxidized in cupric ammonium suspension or in pyridine solution by atmospheric O. This indicates that oxidation of cellulose begins by conversion of the primary OH to aldehyde, which isomerizes with rupture of the cellulose chain.

Text. Research J. Dec. 1946

Solutions of Cellulose

Protective colloid action by cellulose xanthate. Philip C. Scherer. *Rayon Text. Mo.* **27**, 409–14 (Aug. 1946).

Solutions of cellulose in NaOH require less methyl alcohol to bring about precipitation of the cellulose than do mixed solutions of cellulose and cellulose xanthate in NaOH. In the mixed solutions, resistance to precipitation increases as the xanthate content increases while simple cellulose xanthate solutions show slightly decreasing resistance to precipitation with increasing xanthate concentration. It is concluded that cellulose xanthate acts as a protective colloid which prevents agglomeration of the dispersed cellulose. This protective power is independent of the ageing time of the alkali cellulose from which the cellulose xanthate is made, and of the sulfur-cellulose ratio of the particular xanthate used. A substantial amount of supporting data is given. J. F. Keating

Text. Research J. Dec. 1946

Foam Formation and Stability

Foam formation and stability. D. Dervichian. *Corps gras, savons* **2**, 164–71 (1944) (through *Chem. Abstr.* **40**, 5271[4] (Sept. 10, 1946)).

A review with 10 references. It is emphasized that foam phenomena and lowering of the surface tension of liquids are not directly related to each other, but that both are secondary effects due to the presence of a surface-active material.

Text. Research J. Dec. 1946

The Gomberg Reaction

The mechanism of the Gomberg reaction. H. H. Hodgson. *J. Soc. Dyers and Colourists* **62**, 199–202 (July 1946).

Various data connected with the Gomberg reaction are collected and discussed from the viewpoint that, although diaryl formation is explicable on a radical mechanism, the origin of the radicals is in the decomposition of the diazonium ions rather than in that of the isomeric, covalent diazo compounds. The difference in the reaction of diazonium salts with 2-hydroxy-1:4-naphthaquinone in acetic acid and alkaline media, respectively, is readily explainable by modern resonance theory. Author

Text. Research J. Dec. 1946

Properties of Hexanolamine Oleate

Physical-chemical properties of solutions of the colloidal electrolyte hexanolamine oleate. Emanuel Gonick and James W. McBain. *J. Colloid Science* **1**, 127–135 (Mar. 1946).

As the concentration of aqueous solutions of hexanolamine oleate (the 4-methyl, 4-aminopentanol-2 salt of oleic acid) is increased, solutions change from isotropic liquids to solid gels (12% soap) to isotropic liquids (28.8% soap) to a mixture of isotropic and anisotropic phases (28.8 to 39.5% soap) and to homogeneous anisotropic liquids of varying viscosity (39.5 to 80% soap). Freezing point, conductivity, viscosity, and transport data are given for the aqueous solutions. The osmotic coefficients follow Brady's curve for branched chain compounds. The existence of small micelles which contribute very little to the freezing-point lowering but have substantial conductivities is shown by the fact that the conductivity ratio is considerably greater than the osmotic coefficient. The fact that the cationic transport number is negative and that for the anion is greater than unity further demonstrates the presence of conducting micelles which must include hexanolammonium ions. E. D. Klug

Text. Research J. Dec. 1946

Pectin

Effect of methoxyl content of pectin on the properties of high-solids gels. Harry S. Owens and W. Dayton Maclay. *J. Colloid Sci.* **1**, 313–26 (July 1946).

The maximum pH at which high-solids pectin gels can form decreases from 3.5 for purified pectins of 10 to 11% methoxyl to 2.9 for pectins

of 5% methoxyl, providing alkali or possibly acid is used as the catalyst. The maximum pH of gelation is not markedly affected by molecular weight or pectin concentration, but the moduli of rigidity which can be measured reliably increased with the molecular weight. No optimum pH value was observed for pH-moduli curves.
Text. Research J. Dec. 1946 E. D. Klug

Polyethylene

Poly-Ethene. R. Houwink. *Brit. Plastics* **18**, 192–8 (May 1946).

A review of the chemical and physical properties of polyethylene in comparison with polystyrene, guttapercha, and rubber. E. F. Evans
Text. Research J. Dec. 1946

Ramie Fiber

Silver dichroism of micellar systems with fibrous structures. A. Frey-Wyssling and O. Wälchli. *J. Polymer Sci.* **1**, 266–74 (Aug. 1946).

A study of the crystal structure of silver deposited in ramie fiber.
Text. Research J. Dec. 1946 E. D. Klug

Sedimentation Velocity of Macromolecules

The sedimentation velocity of long-chain macromolecules. J. J. Hermans. *Rec. trav. chim.* **63**, 219–25 (1944) (in German) (through *Chem. Abstr.* **40**, 4938[5] (Sept. 10, 1946)).

Burgers' formula for the sedimentation velocity of spherical particles (*C.A.* **37**, 3652[8]) was modified to apply to randomly-kinked long-chain molecules. The velocity is inversely proportional to the η of the medium and the length of a monomeric unit and directly proportional to the force on a monomeric unit and the factor $(1 + N^{\frac{1}{2}})$, where N is the number of statistical fiber elements. This is in agreement with experimental data (from the literature) for 2 of the 3 cases examined. If N is large, $(1 + N^{\frac{1}{2}})$ approaches $N^{\frac{1}{2}}$, which is the proportionality factor introduced by Kuhn and Kuhn (*C.A.* **38**, 673[9]) for the case of tightly coiled molecules that do not permit free flow of liquid through the chains. The theoretical treatment of K. and K. is in accord with H.'s views. It is emphasized that the hydrodynamic interaction in tightly coiled molecules is much less effective in the presence of a strong velocity gradient. For this reason, some polymer solutions conform to Staudinger's η rule, although the sedimentation constants vary with molecular weight.
Text. Research J. Dec. 1946

Viscosity of Fiber Molecules

An explanation by means of a model of the inner viscosity (the form viscosity constant) of fiber molecules. I. Werner Kuhn and Hans Kuhn. *Helv. Chim. Acta* **29**, 609–26 (1946) (in German); cf. *C.A.* **40**, 3327[1] (through *Chem. Abstr.* **40**, 4937[9] (Sept. 10, 1946)).

The dependence of the inner viscosity on restricted rotation within the fiber molecule is studied on the basis of a statistical model. The square of the change, due to rotation, of the distance, Δh, between the two ends of a fiber molecule dissolved in a solute having a vanishingly small viscosity is proportional to the time and, in a homologous polymer series, is proportional to the degree of polymerization. The time in which Δh amounts to twice the average distance between the ends of a statistically coiled molecule is called the macro time of configuration change and is independent of the degree of polymerization. A diffusion constant applied to the movement of the ends of the molecule is proportional to the degree of polymerization and is also proportional to the form viscosity constant. The form viscosity constant divided by the degree of polymerization is a constant for a homologous polymer series. The excitation energy connected with the restricted rotation is calculated from measurements of viscosity and streaming double refraction. The excitation energy in calories per mole is 11,000 for nitrocellulose in butyl acetate, 8,500 for nitrocellulose in cyclohexanone, 7,600 for methylcellulose in water, and 11,400 for polystyrene in cyclohexanone.
Text. Research J. Dec. 1946

Viscosity-Molecular-Weight Relationship

Relation between molecular weight and intrinsic viscosity of polymethyl methacrylate. J. H. Baxendale, S. Bywater, and M. G. Evans. *J. Polymer Sci.* **1**, 237–44 (Aug. 1946).

In the polymerization of methyl methacrylate with ferrous ion-hydrogen peroxide, the $\log [\eta]$ was found to be proportional to $\log (2\Delta m/a)$ where $[\eta]$ is the intrinsic viscosity of the polymer, Δm is the moles/liter of monomer polymerized and a is the moles of hydrogen peroxide/liter used. It follows that one molecule of hydrogen peroxide is required to initiate and one molecule is required to terminate each polymer chain. Polymer, prepared with ferrous ion-hydrogen peroxide, was fractionated and the average chain length of the fractions was determined osmometrically. From these data and intrinsic viscosities, both measured in benzene, the constants K and α in the expression $n = K[\eta]^{\alpha}$ where n is the chain length were determined. For benzene solution $n = 2810[\eta]^{1.32}$. Using data of Schultz on polymers prepared with benzoyl peroxide, the equation in $CHCl_3$ solution was calculated to be $n = 1810[\eta]^{1.22}$. It is concluded that there is little if any difference between polymers prepared with ferrous ion-hydrogen peroxide and benzoyl peroxide. E. D. Klug
Text. Research J. Dec. 1946

Reaction of Sulfur with Olefins

Modern views on the chemistry of vulcanization changes. I. Nature of the reaction between sulfur and olefins. E. Harold Farmer and F. W. Shipley. *J. Polymer Sci.* **1**, 293–304 (Aug. 1946).

Unaccelerated reaction between sulfur and monolefins like cyclohexene gives cross-links almost entirely. With diolefins like dihydromyrcene, intramolecular cross-linking, i.e., cy-

clization, occurs mainly, with some intermolecular polysulfide cross-linking. A free-radical mechanism is suggested. Experimental details are omitted. E. D. Klug

Text. Research J. Dec. 1946

Reaction of Olefins with Hydrogen Sulfide

Modern views on the chemistry of vulcanization changes. II. Role of hydrogen sulfide. Ralph F. Naylor. *J. Polymer Sci.* **1**, 305–11 (Aug. 1946).

The addition of H_2S to olefins is catalyzed by small amounts of sulfur, but the rate of addition is too slow to provide a satisfactory basis for an H_2S-actuated mechanism of vulcanization. The main products with polyisoprenes are substituted pentamethylene sulfides, derived by intramolecular normal addition of the initially formed monomercaptans. In the presence of ultraviolet light, the main products are monomercaptans and substituted pentamethylene sulfides. The mechanism of vulcanization is discussed in light of these data. Experimental details are omitted.

Text. Research J. Dec. 1946 E. D. Klug

Chemistry of Vulcanization

Modern views on the chemistry of vulcanization. III. Reaction of sulfur with squalene and with rubber. George F. Bloomfield. *J. Polymer Sci.* **1**, 312–17 (Aug. 1946).

Squalene which contains 6 isoprene units reacts with sulfur in much the same way as the di-isoprene, dihydromyrcene, in that intramolecular sulfide linkages as well as intermolecular polysulfide linkages are formed. The action of sulfur on rubber in the absence of accelerators, etc., seems to be the same. With accelerators, sulfur reacts with the thiol groups of mercaptans, forming di- and polysulfides and H_2S. In the presence of rubber, this reaction occurs without the liberation of H_2S; it appears that thiol groups are intermediate in the formation of di- and polysulfide cross-links. Experimental details are omitted. E. D. Klug

Text. Research J. Dec. 1946

Starch-Fatty Acid Complex

The complexes of fatty acids with amylose. F. F. Mikus, R. M. Hixon, and R. E. Rundle. *J. Am. Chem. Soc.* **68**, 1115–23 (June 1946).

The method of Schoch and Williams (*J. Am. Chem. Soc.* **66**, 1232) was used to obtain amylose-fatty acid precipitates. Amyloses with a helical chain configuration will bind fatty acids whereas amylose with an extended chain configuration will not. Bound fatty acids may be extracted with methanol but not with carbon tetrachloride. The fatty acids, like iodine, are held in the holes in the center of the helical amylose tubes, not in the interstices between these tubes. This structure of the complex explains quantitatively the fatty-acid content of the complex, the effect of previous fat content on the binding power of the defatted granules, the low fat content of waxy starches, the differences between many solvents in fat extraction, and the inability of amylopectin to bind fatty acids. The force between the amylose and the complexing agent is explained in terms of dipolar interactions, adopting the mechanism suggested for the iodine complex (*J. Am. Chem. Soc.* **65**, 1707).

A. R. Macormac

Text. Research J. Dec. 1946

Chlorine Derivative of Starch

The chlorination of starch with phosphorus pentachloride. H. N. Barham, E. S. Stickley, and M. J. Caldwell. *J. Am. Chem. Soc.* **68**, 1018–24 (June 1946).

Pentachlorostarch was prepared as follows: 40 g. of corn starch dried for 12 hrs. *in vacuo* at 100°C was mixed with 540 g. dry, finely ground phosphorus pentachloride and heated in 3 steps, keeping anhydrous condition throughout. The 1st step was 15 to 17 hrs. at 100–105°C. The 2nd was about 24 hrs. at 125–135°C and the 3rd was 160–170°C for 8 hrs. The completion of the different stages could be determined by the appearance of the mass. The final residue was treated with ice water to remove the residual phosphorus pentachloride and then scrubbed with cold water. The pentachlorostarch was a yellow-to-orange amorphous solid. Chemical evidence leads to the conclusion that chlorine atoms are on carbons 2, 3, 5, and 6 of the glucose ring; the 5th atom is probably on carbon 1. At least one of the chlorines has replaced a hydrogen.

A. R. Macormac

Text. Research J. Dec. 1946

Action of Bisulfite on Wool

The chemical constitution and physical properties of bisulphited wool. E. G. H. Carter, W. R. Middlebrook, and H. Phillips. *J. Soc. Dyers and Colourists* **62**, 203–11 (July 1946).

Evidence is surveyed for the existence of 4 subfractions of the cystine-*S* of wool (*A*, *B*, *C*, and *D*) which differ in their reactivity towards sodium bisulfite and alkalis. These 4 subfractions fall into 2 approximately equal main fractions $(A + B)$ and $(C + D)$; $(A + B)$ gives thiol and *S*-cysteine-sulfonate groups with bisulfite; $(C + D)$ does not react with cold bisulfite, but under the action of hot bisulfite decomposes and yields combined α-aminoacrylic acid. With alkalis, fraction $(A + B)$ gives combined lanthionine, whereas fraction $(C+D)$ again gives combined α-aminoacrylic acid. Fibers the $(A + B)$ fraction of which has been broken do not supercontract until after they have been boiled; they then supercontract even when boiling causes the disulfide cross-linkages to reform. Even when boiled, the fibers of which only subfraction *B* has been broken will not supercontract. After fibers have been boiled in sodium bisulfite solution, supercontraction cannot be prevented by rebuilding fraction $(A + B)$. Evidence is discussed in favor of the view that the chief factor in the stability of keratin and other fibrous proteins is main-chain adhesion. Polar side chains are potential sources of weakness since they can give rise to swelling and hydration. In the keratins their activity is suppressed

by the proximity of disulfide cross-linkages. Authors

Text. Research J. Dec. 1946

Binding of Copper by Wool

Combination of wool protein with heavy metal salts as a function of pH. I. Copper sulfate. K. S. LaFleur. *Am. Dyestuff Reptr.* **35**, 383–6, 395–7 (Aug. 12, 1946).

The copper content of wool swatches in equilibrium with copper sulfate baths of varying acidity was found to increase with increasing pH by a method depending upon the quantitative deposition of the copper present upon a platinum cathode in a strongly acid electrolytic bath. It is judged that with rise in pH the increasingly dissociated "free" carboxyl groups in wool constitute the determining factor in the binding of copper, which is in this case apparently totally cationic. The usual wool-amino group—sulfate combination also takes place. A number of experiments which were performed in an attempt more adequately to describe the wool-copper sulfate-sulfuric acid-water system, as part of a study of the dissociation curves of wool protein as a function of pH, are described.

K. S. Campbell

Text. Research J. Dec. 1946

BLEACHING: DYEING: FINISHING

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Nylon Dyeing

Some aspects of nylon dyeing. A. K. Saville. *Am. Dyestuff Reptr.* **35**, P58–9 (Jan. 28, 1946).

A valuable review of the current status of nylon dyeing; recent efforts to improve the quality of the results obtainable in dyeing nylon are discussed. Of particular interest are the pad-steam process and the closed-jig method with acid colors, use of vat colors at high temperatures, and naphthol dyes applied by the two-bath method. While acetate dyes are still of greatest general importance for nylon, selected members of other classes have specific fastness advantages and these will probably increase in number. For example, underwear fabrics are likely to be dyed with vat colors, while azoic colors are well suited for men's half hose, because of good wash-fastness properties despite the poor light-fastness of both of these dyes on nylon.

K. S. Campbell

Text. Research J. Dec. 1946

Dyeing Polyamides

The action of polyamides during dyeing. II. E. Elöd and Th. Schachowskoy. *Melliand Textilber.* **25**, 237–42 (1944); cf. *C.A.* **38**, 2493[5] (through *Chem. Abstr.* **40**, 4886[6] (Aug. 20, 1946)).

In all polyamides tested dye absorption depended on the pH of the bath with the possible exception of one grade, Igamid U. This dependence is not due to the increased or decreased swelling of the material. Polyamides differed in the amounts of dye taken up. With 6 hrs.' dyeing Igamids 6A, IC, and 85B showed much greater dye-acid absorption than did Igamids B, A, and U. It appears that those polyamides which absorb more water show likewise larger dye-acid-absorption powers. It is concluded that the difference in the dye-acid absorption between the various products depends on various structural properties of the different polyamides as well as other factors. The dependence of dye-acid absorption on the concentration of the dye liquor is the more noticeable with those polyamides which absorb the larger amounts of dyes. At H-ion concentrations above 0.1 *N*, the dye-acid absorption increases appreciably for all polyamides. This increase of the dye-acid absorption is not due entirely to the polyamide degradation that occurs in the highly acid medium. The dyeing properties of all polyamides seem to differ from those of proteins, such as wool and silk, which reach a saturation value of dye absorption at a definite pH value. The examination of the dye-acid discharge of dyed polyamide in water which was adjusted to the same pH value as that of the final pH of the bath showed that generally polyamides with larger dye-absorption powers also give up the dye more rapidly. Exceptions to this rule are Igamid IC and B. On all polyamides dyes are discharged further and faster than on wool. **III.** *Ibid.* **309–14.** Experimental data are given on the dependence of the dye-acid absorption of polyamides on the length of the dyeing period, the influence of the degree of degradation of polyamides on the dye intake, the temperature dependence of the dye intake, and the concentration dependence of the dye-acid intake. The polyamides differ in velocity of dye-acid absorption and in their equilibrium condition. Although Igamid A, B, and 6A are nearly alike in molecular size they take up different amounts of dye acid. The polyamides are degraded during the dyeing, but this does not increase dye-acid absorption. For the absorption of the dye acid those groups in polyamides are significant whose number is changed little or none by the degradation. Evidently the degree of polymerization plays no decisive role in the dye-acid absorption at the pH values used. At 85°, 90°, and 95°, Igamid 6A absorbs approximately the same amount of dye at the state of equilibrium while Igamids A and B even after 48 hrs. of dyeing still differ in dye-acid absorption. From the concentration dependence of the dye-acid absorption, as well as from the fact that Henry's law does not apply, it was concluded that the reciprocal action between the dye acid and the polyamides is caused by intermolecular forces.

Text. Research J. Dec. 1946

Photographic Screen Printing

Screen printing. I. Screen printing as done in textile mills of the Baltic republics. I. Shteĭn. *Tekstil. Prom.* **6**, No. 1, 20–3 (1946) (through *Chem. Abstr.* **40**, 5567[9] (Sept. 20, 1946)).

The pattern on the silk screens used in these mills is made by means of photosensitive substances. The silk is coated with gelatin and photosensitized with $K_2Cr_2O_7$ or $(NH_4)_2Cr_2O_7$. A layer of varnish may or may not be interposed between the

silk and the gelatin. The pattern is transferred onto the gelatin photographically. Details of preparation of the screen, its use, and preparation of the printing dyes are described.
Text. Research J. Dec. 1946

Screen printing. II. Photographic printing. A. Frolov. *Ibid.*, 23 (through *Chem. Abstr.* **40**, 5568[6] (Sept. 20, 1946)).

Screen printing in which the screen is prepared as a photographic film is described as practiced in the Moscow silk-finishing plants.
Text. Research J. Dec. 1946

Rotproofing

The dyeing of cotton with mineral khaki. Part VII—The fungicidal and bactericidal efficiencies of cotton yarn treated by various mineral khaki processes. E. F. Race, F. M. Rowe, and J. B. Speakman. *J. Soc. Dyers and Colourists* **61**, 310–21 (Dec. 1945).

To determine what factors were of most importance in moldproofing cotton treated with chromium and iron compounds, yarns were treated with 7 types of chromium-iron pigments and subjected to a variety of tests for fungicidal and bactericidal efficiencies. It was found that the only toxic principle in chrome-iron-treated yarns was hexavalent chromium; this is produced only by the chromate development process and is not permanent, being hydrolyzed by water and reduced by the cellulose of the material; alkali-soluble trivalent chromium plays no part in rotproofing. Waterproofing with a paraffin wax after-treatment imparts a transient resistance to attack by fungi and bacteria. Moldproofing efficiencies were evaluated by 5 types of tests; viz., a deep, vertical soil-burial test, a horizontal burial test in petri dishes, a test in which yarn was placed on the surface of a horse-dung preparation, a horse-dung inoculation followed by incubation on filter candles or in glass jars, and a pure culture inoculation. It appears that for a comparison of moldproofs which do not differ widely in their efficacy it is necessary to employ a pure culture inoculum with a slight amount of nutrient medium provided. The testing technique employed should be chosen, taking into account expected service-life environmental conditions.
Text. Research J. Dec. 1946 K. S. Campbell

FIBERS: YARNS: FABRICS: MECHANICAL PROCESSES

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Bast Fibers

Structure, properties, and preparation of certain bast fibers. Harriette Brita Hanson. *Iowa State College J. Sci.* **20**, 365–83 (1946) (through *Chem. Abstr.* **40**, 4888[5] (Aug. 20, 1946)).

A study was made of the relative quantity, properties, and extractability of the fiber of the plants: (1) *Apocynum cannabinum*, Indian hemp or dogbane; (2) *Cannabis sativa* L., marihuana hemp; (3) *Asclepias syriaca* L., common milkweed; (4) *Asclepias sullivanti* Engelm., smooth-leafed milkweed; (5) *Asclepias verticillata* L., a whorled milkweed; (6) *Gonolobus laevis* Michx., climbing milkweed; (7) *Polygonum scandens* L., climbing false buckwheat; and (8) *Linum usitatissimum* L., flax. The quantity of fiber in 1, 2, 3, 4, and 6 justified further study to devise means for economical commercial production. Fiber cells of 1, 2, 3, and 4 were sufficiently similar to flax fibers to be spun and woven into cloth. Fibers of 1, 2, 3, 4, and 6 react to reagents used in the care of fabrics much as flax fibers react. The luster and high quality of fiber in 6 merits study in its cultivation, processing, and utilization. Processes for retting on a laboratory scale are reported.
Text. Research J. Dec. 1946

Fungal Damage to Cotton

Fungal damage to sun-exposed cotton duck. Robert K. Zuck and Wm. W. Diehl. *Am. J. Botany* **33**, 374–82 (1946) (through *Chem. Abstr.* **40**, 4892[8] (Aug. 20, 1946)).

Certain angiocarpous fungi heretofore unrecognized as being primarily involved in the decay of gray duck or in the decomposition of cellulose are here reported as significant agents in the slow cellulosic breakdown of fabric during aerial exposure. They are *Diplodiella cowdellii*, *Hendersonia sarmentorum*, *Hendersonia* sp., *Leptosphaeria* sp., *Diplodia* sp., *Phoma herbarum*, and *Ophiobolus* sp. These fungi have tended to escape detection for 2 reasons: the employment of standard plating techniques which do not detect these slow-growing fungi; samples of gray duck develop these angiocarps only after long periods of aerial exposure. A quantitative method is given for evaluating fungal damage as distinct from nonbiological damage when these occur simultaneously on the same fabric.
Text. Research J. Dec. 1946

Effect of Heat on Nylon

Some effects of dry heat upon the properties of nylon fabric. Philadelphia Section A.A.T.C.C. *Am. Dyestuff Reptr.* **35**, P38–42 (Jan. 14, 1946).

The effect of varying the time and temperature of application of dry heat to nylon fabric, relaxed and under tension, on its strength, elongation, and dyeing properties is shown graphically. A temperature near 400°F appears to be critical in many respects, degradation becoming marked after 10 secs.' exposure beyond this point. Dry heating may either increase or decrease the subsequent absorption of dye depending upon the dye used; Cloth Fast Blue R (Ciba) is absorbed to about 3 times the normal extent when the fabric has been heated to 470°F for 15 secs., and it is suggested that this dye may be of value as an indicator of heat damage in nylon. K. S. Campbell
Text. Research J. Dec. 1946

Degreasing Fabrics

The degreasing of fabrics. Arnold Lassieur. *Corps gras, savons* **2**, 68–71 (1944) (through *Chem. Abstr.* **40**, 5569[9] (Sept. 20, 1946)).

A sample of cloth charged with a known quantity of alimentary fat

was washed, and the fat content of the cloth, after drying, was determined. A piece of cotton charged with a mixture of tallow and grease and washed at 90° with arachidic soap lost 23% of its grease content after 1 min. and 28% after 2 min. Longer washing was without effect. Repeated washings and dryings did not eliminate any additional greasy substance. Brushing between the washings, in order to change the surface texture, had only a very limited degreasing effect. Na_2CO_3 solution (4 g./l.) removed 28% of the grease from cotton after 3 5-min. washings at 90°. Brushing in between increased the cleansing index to 38%. Rayon soiled and cleaned in the same manner was freed of 82% fat after 5 washings (the 1st washing eliminating only 37%). The percentage of grease eliminated by washing depends on the material, the quality and quantity of grease, the detergent, the temperature, and the mechanical action. Degreasing by washing is always incomplete, while mineral pigments are completely eliminated. Na_2CO_3 degreases better than soap. A kitchen towel laundered in the customary manner with Na_2CO_3 and soap was freed of 40% of its grease (determined by ether extraction); this fact showed that laundering is not identical with degreasing.

Text. Research J. Dec. 1946

Glyoxal Stabilization Process

Recent developments in rayon fabric stabilization. Jack Epelberg. *Am. Dyestuff Reptr.* **35**, 343–6 (July 15, 1946); *Rayon Text. Mo.* **27**, 355–6 (July 1946) and 405–6 (Aug. 1946).

Stabilization of rayon fabrics to shrinkage is accomplished by treatment with a water solution of glyoxal and an acid catalyst. The fabric is padded with this solution, then dried. It is then baked to react the glyoxal with the cellulose, followed by a wash to remove the unreacted chemicals. The fabric is then slack-dried and steamed to width on a tenter frame. The glyoxal process is not satisfactory for acetate rayon, but is for blends of acetate and viscose where the latter predominates. It is not satisfactory for cotton because of the sensitivity of cotton to the acid catalyst at high temperatures. Chlorine retention is absent in fabrics so treated. The process has little effect on vat colors but changes many of the direct colors. No change in hand or texture occurs and the finish is permanent to washing. Some decrease in tensile strength is noted. J. F. Keating

Text. Research J. Dec. 1946

Spindle Adapter

Aluminum adapter for spindles. Anon. *Am. Wool and Cotton Reptr.* **60**, 34 (Sept. 26, 1946).

A report on the conversion of the old type of warp spinning to large packages by means of aluminum adapters to fit onto the old spindles so that paper tubes may be used. It is said that Joanna Textile Mills, where the adapters were developed, have increased the amount of yarn put onto a bobbin by 34% by going from a $6\frac{7}{8}$-in. traverse to an $8\frac{7}{8}$-in. traverse with a 2-in. ring at a cost of about $1.00 per spindle.

H. J. Burnham

Text. Research J. Dec. 1946

WAR RESEARCH ON TEXTILES*

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U. S. Research

Adhesion Test

Determination of adhesion of plies of coated fabrics. Progress report. (Rept. 19.) U. S. Quartermaster Depot, Jeffersonville, Indiana. PB 39777, Aug. 1944; 14 pp.; microfilm, $1.00—photostat, $1.00 (through *Bib. Sci. and Ind. Reports* **3**, 203 (Oct. 18, 1946)).

Examination shows that coated fabrics of different types require application of different techniques in the determination of adhesion of coating to base cloth. A suitable method is described in this report for performing the adhesion test on fabrics for 2-man mountain tents. The question of the value of an adhesion test is raised in connection with the durability of coated fabrics. A preliminary estimate points to the conclusion that superior initial adhesion in the 2-man mountain-tent fabric does not presage good resistance to flaking or peeling during the life of the material. Tables and graphs are included. An appendix presents proposed method for the determination of adhesion of vinyl-resin coatings to base fabrics.

Text. Research J. Dec. 1946

Strength Testing

A study of the effect of reducing the rate of traverse from 12 to 6 inches per minute in the grab method for determining the break strength of cotton duck. U. S. Quartermaster Depot, Jeffersonville, Indiana. PB 37983, Feb. 1944; 10 pp.; microfilm, $1.00—photostat, $1.00 (through *Bib. Sci. and Ind. Reports* **3**, 128 (Oct. 11, 1946)).

Experiments with yarns had indicated that the dispersion or standard deviation of data observed in tests made to determine the break strength of fabrics is narrowed by reducing the rate of traverse from 12 in. to 8 in. per min. This study was undertaken to ascertain whether a significant reduction in standard deviation is obtained with cotton duck, when a similar reduction in rate of traverse is made in the grab method of determining break strength. The tests, as reported here, show that no practical change occurs in the standard deviations of the test data for cotton ducks, ranging from 7.90 oz. per sq. yd. to No. 6. Tables and a short bibliography are included in the report.

Text. Research J. Dec. 1946

A study of the grab method for determining the break strength of cotton duck. U. S. Quartermaster Depot, Jeffersonville, Indiana. PB 37982, Dec. 1943; 13 pp.; microfilm, $1.00—photostat,

* Copies of the original reports abstracted in this section may be obtained from the U. S. Department of Commerce, Office of Technical Services, Washington 25, D. C.

$1.00 (through *Bib. Sci. and Ind. Reports* **3**, 128 (Oct. 11, 1946)).

Contiguous grab samples of No. 6 cotton duck were arranged in series, and the various series tested for break strength, 2 under standard conditions, the others under conditions differing from the standard by known variations. Table I presents a summary of results and Table II shows a comparison of variation effects. This study shows that the grab-sample method of determining the break strength of textiles is subject to variations which result in statistically significant differences in the arithmetic average. The effects of variations in jaw conditions, types of testing machines, ranges on the same machine, faces of the fabric, alignment of the grab sample, cupping of the sample around the jaws, test operators, and the relative position of the grab samples in the original fabric are included in the study. The effect of variation in relative position is used as the norm in judging all other effects. All but 2 of these variations in the method produce small, but statistically significant, effects upon the result. Large differences in results are obtained by different operators of the test equipment. The only perceptible pertinent difference between operators was that of physical strength, which is required to operate the instrument.

Text. Research J. Dec. 1946

German Research

Cotton, Rayon, and Silk

A survey of the German cotton, rayon, and silk industries. (BIOS Final Rept. 574, Item 22, 31.) D. W. Hill and others. PB 34729, n.d.; 168 pp.; microfilm, $4.00—photostat, $12.00 (through *Bib. Sci. and Ind. Reports* **3**, 57 (Oct. 4, 1946)).

The chief textile raw material now being processed in Germany is rayon, both filament and staple. The firms processing it fall, broadly speaking, into 2 groups—those which have approached the task from the silk industry and use a high proportion of filament rayon and those which have approached it from the cotton industry and use mostly rayon staple. The approach from the silk industry is revealed by the use of sectional warping, high-quality, continental-type looms, and individual methods of finishing; while that from cotton is shown by warp preparation on the beaming and tape-sizing system, the more frequent use of the Lancashire type of loom, and bulk methods of finishing such as are usually employed in Lancashire for processing fabrics woven from staple yarns. However, the growth of an industry concerned with rayon alone, with methods of processing that are related to the particular characteristics of the fibers, may be clearly discerned. Part I of this report covers spinning and weaving; dyeing, printing, and finishing; and research and control of processes. Much of the research and testing in the textile field has already been described in reports listed under this section. With the exception of the I. G. laboratories, there was very little evidence of any considerable body of fundamental research in spinning, weaving, or finishing. Part II gives a list of targets visited, with a brief description of each factory, and the numbers and types of machines in the more important works. Photographs and diagrammatic drawings of various machines, as well as specimens of German utility specifications, are included.

Text. Research J. Dec. 1946

Processing Rayon Staple

The processing of rayon staple fiber on cotton-spinning machinery in Germany. (BIOS Final Rept. 634, Item 22, 31.) W. A. Hunter and others. PB 41235, Mar. 1946; 52 pp.; microfilm, $2.00—photostat, $4.00 (through *Bib. Sci. and Ind. Reports* **3**, 202 (Oct. 18, 1946)).

Most of this report deals with a visit to Lehrspinnerei, Denkendorf, an experimental spinning plant built and equipped during 1937–8 for the purpose of testing new machines on mill scale, and for developing new devices to improve the performance and production of machinery designed to process staple rayon and mixtures of rayon with other fibers, animal and vegetable. Drawings illustrate the layout of the plant, which contains 5,000 spinning spindles on the cotton system and 3,000 spinning spindles on the worsted system with all the necessary preparation machinery. A sketch of the institute as originally planned is included. Short reports on the following plants engaged in spinning yarns from rayon staple fiber on existing cotton-spinning machinery are included: Süddeutsche Spinnerei, Kuchen; Germania Spinnerei, Epe, Gronau; Niehues and Duetting, Nordhorn; and Ludwig Povel, Nordhorn. Machinery and processes are described and drawings and graphs are included.

Text. Research J. Dec. 1946

Wool-Combing Industry

German wool-combing industry and wool-grease-extraction processes. Jan.-Feb. 1946. (BIOS Final Rept. 474, Item 22, 31.) Geoffrey M. Robertshaw. PB 34745, July 1946; 70 pp.; microfilm, $2.00—photostat, $5.00 (through *Bib. Sci. and Ind. Reports* **2**, 994 (Sept. 27, 1946)).

This report summarizes the information gained during the investigation of the German wool-combing industry by stating that from a technical point of view little new information has been discovered. A visit to the works of Schachenmayr, Mann & Co., at Salach (Würt), disclosed a substitute for leather on the preparing boxes, combs, and finishers. The material was supplied in rolls by I. G. Farben, and cemented into the required sizes. The life of these substitutes was stated to be equal to, if not better than, that obtained from leather. The process of cementing and making up of these leathers was regarded as secret and the plant was well guarded. The extraction of wool-grease products falls into two categories: (1) works where grease-extraction is carried out on extremely unscientific and antiquated lines; and (2) works where an elaborate plant for the benzene-extraction process has been installed. A list is given of chemical engineers

and dealers who have supplied materials to grease-extraction plants in the British Zone of Germany. Detailed reports of visits to the following plants are presented, including data on processing and types of machines in use, and, in some cases, investigation of the methods of extraction of wool grease from wool-scouring liquor: Wollwäscherei & Kammerei, Döhren bei Hannover; Hamburger Wollkämmerei G.m.b.H., Harburg-Wilhelmsburg; Bremer Wollkämmerei, Blumenthal (Unterweser); Kammgarnspinnerei Delmenhorst G.m.b.H., Delmenhorst; Schoellersche Kammgarnspinnerei Eitorf A.G., Eitorf (Rheinland); Schachenmayr, Mann & Co., Salach (Würt); and Merkel & Kienlin, G.m.b.H., Esslingen. Diagrams of plant layouts are included.
Text. Research J. Dec. 1946

PATENT REFERENCES

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Dyeing Cellulose Acetate

Coloration of textile materials. Herbert Platt and Cyril M. Croft (to Celanese Corporation of America). U. S. 2,405,669 (Aug. 13, 1946).

An inclosed jig is used to dye acetate fabrics with high-temperature dyes. By this means, the fabric on the rolls is maintained at the dye-bath temperature. Uneven dyeing is thus avoided. The claims cover the method but not the machine. A. R. Martin
Text. Research J. Dec. 1946

Apparatus for Treating Fibers with Liquids

Apparatus for applying treating liquids to fibers. Hughes L. Siever (to Borne Scrymser Company). U. S. 2,405,857 (Aug. 13, 1946).

A moving lap of fibers is sprayed with solution from a series of nozzles. By means of vibrating the nozzles, small droplets of liquid are obtained.
Text. Research J. Dec. 1946 A. R. Martin

Bonded Web or Sheet Material

Manufacture of artificial fibrous sheet material. Robert Pickles and John Pickles. U. S. 2,405,978 (Aug. 20, 1946).

Flock, ranging in length from 2–10 ml., is treated on a traveling conveyor with a bonding agent and thence through pressure rolls to form a sheet material. A. R. Martin
Text. Research J. Dec. 1946

Polyacrylonitrile Polymers

Ray Clyde Houtz, George Henry Latham, Arthur O. Rogers, V. L. Hansley, William Hale Charch, and Theron G. Finzel (to E. I. du Pont de Nemours & Co., Inc.). U. S. 2,404,713–728, inclusive (July 23, 1946).

This group of patents covers the preparation spinning solutions of polyacrylonitrile, or of copolymers containing at least 85% polyacrylonitrile, using various solvents and means of obtaining stable solutions. A. R. Martin
Text. Research J. Dec. 1946

Vinyl Polymers

Vinyl polymers. Harold W. Arnold, Merlin Martin Brubaker, and George L. Dorough (to E. I. du Pont de Nemours & Co., Inc.). U. S. 2,404,781 (July 30, 1946).

A process for the preparation of vinyl polymers in the presence of salts of perdisulfuric acid.
Text. Research J. Dec. 1946 A. R. Martin

New Vinyl Polymer

Production of copolymers of monoethenoid compounds. Daniel E. Strain (to E. I. du Pont de Nemours & Co., Inc.). U. S. 2,404,817 (July 30, 1946).

Method is disclosed for producing copolymers of such materials as unsymmetrical dichloroethylene and methyl methacrylate.
Text. Research J. Dec. 1946 A. R. Martin

Vinyl Copolymers

Process for the conjoint polymerization of vinyl chloride with fumaric esters. Harold W. Arnold (to E. I. du Pont de Nemours & Co., Inc.). U. S. 2,404,780 (July 30, 1946).

Vinyl chloride is copolymerized with esters of fumaric acid. A. R. Martin
Text. Research J. Dec. 1946

MISCELLANEOUS

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Review of 1943–4 Literature

Fibers and textiles. W. Knight. *Ann. Rept. Progress Rubber Technol.* **8**, 71–3 (1944); cf. *C.A.* **39**, 1762[5] (through *Chem. Abstr.* **40**, 6262[5] (Oct. 20, 1946)).

A review of periodical and patent literature of 1943–4, with 30 references.
Text. Research J. Dec. 1946

Italy's Textile Industry

Italy's textiles: vital factor in rehabilitation. Mary E. Poole. *Foreign Commerce Weekly* **23**, 8–11, 52–3 (June 1, 1946).

Eighty percent of Italy's textile industry is located in Lombardy and Piedmont, in northernmost Italy, but the industry is also scattered throughout the country. It employed 470,000 persons and an additional 380,000 were employed in the clothing and accessory industry before the war. In addition, there is considerable handicraft production of laces, carpets, and tapestries. The textile industry was highly efficient before the war and its equipment is still 95% intact, but it is badly in need of a revival of imports of cotton and wool, both requiring a revival of foreign trade. R. B. Evans
Text. Research J. Dec. 1946

Textiles in Russia

The Soviet textile industry. Lev Karasin. *Text. Weekly* **38**, 112 (July 19, 1946).

The new 5-year plan as applied to textiles aims to provide Russia with 3,300,000 spindles and 65,000 looms by 1950. This year alone the mills will get 210,000 spindles. It is said that particular attention is being paid to the production of the latest improved types of labor-saving textile machinery and that one machinery-building plant in Moscow will soon be turning out 22 different types of equipment, one of which is a complicated wool card weighing 24 tons. H. J. Burnham
Text. Research J. Dec. 1946

Textile Research Journal

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